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C. H. CLARK, *Chairman* — PROF. E. R. SCHWARZ

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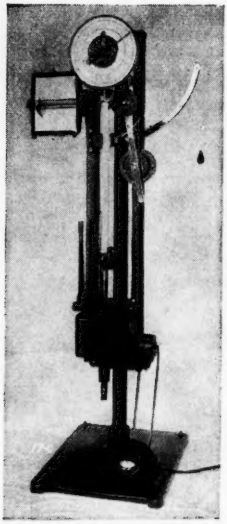

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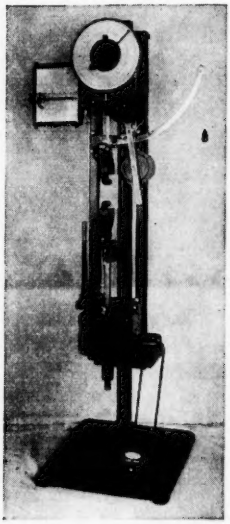

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The Editor's Page

THIS page has been reserved heretofore for Mr. Charles H. Clark, who has been Editor of the magazine and Secretary of the Institute since its formation. It is with much regret that the Directors have been obliged to accept and announce Mr. Clark's resignation because of prolonged illness. As this is being written, Mr. Clark's condition is regarded as being favorable and all of his many friends sincerely wish a speedy and complete recovery for him.

The Institute has never been burdened with an over-supply of financial resources and much credit is, therefore, due Mr. Clark for what he has accomplished under adverse conditions and with inadequate equipment. The Directors of the Institute now have under consideration suggested plans for the future conduct of the Institute affairs.

Luncheon and Research Conference

At a special meeting of the Board of Directors held October 31, 1940, it was decided to postpone the annual business meeting and election of officers scheduled for November 14th until some future and as yet undetermined date.

This is to afford time for study of plans for the future of the Institute.

The Research Conference will be held as announced on Thursday, November 14, 1940, at the Hotel Pennsylvania, New York, N. Y., at 12:30 p.m. It will begin with a luncheon open to members and guests immediately following which there will be the most important technical meeting ever held under Institute auspices. Important research projects will be presented and discussed. Mr. W. D. Appel, Chairman Research Council, will be in charge of the conference.

1. **Effects of Processing on the Wool Fibre.**
   Leader: Mr. Rinaldo A. Lukens, Chairman, Administration Committee.
   The program for this new project will be presented and plans for the work will be discussed.
2. **Textile Drying Research.**
   Leader: Dr. A. C. Walker, Chairman, Administration Committee.
   The outcome of the project now being completed will be discussed and the desirability of organizing a new research relating to drying will be considered.
3. **Warp Sizing Research.**
   Leader: Mr. Carl R. Harris, Chairman, Administration Committee.
   Progress in this project will be reported.
4. **Economic Research.**
   Leader: Mr. Fessenden S. Blanchard, Chairman, Administration Committee.

Effect of Light and Heat on Color and Deterioration of Viscose, Acetate, and Cuprammonium Fabrics*

By HAZEL FLETCHER and MIRIAM H. HOUSTON†

Summary

Light decreased the breaking strength of the viscose, acetate and cuprammonium rayons uniformly. Heat decreased the breaking strength of all of them considerably, but the cellulose acetate rayons were more resistant to heat than the other two kinds. Light produced little change and heat much change in color. The cellulose acetate rayon fabrics were affected the least. The ash alkalinity showed no perceptible increase or decrease for any of the rayons exposed to light or heat. The amount of increase in the copper number and per cent solubility in sodium hydroxide was approximately the same for the viscose and cuprammonium rayon fabrics, and it was less in the case of the cellulose acetate rayon fabrics. Thus the greater change in breaking strength, copper number and per cent solubility in sodium hydroxide indicates that the viscose and cuprammonium rayon fabrics were deteriorated more by heat than the cellulose acetate rayon fabrics.

Introduction

THE effects of light and heat upon the natural fibres have been studied extensively, but little is known as to their effects upon rayon. Results of studies which have been published on the tendering of the rayons by light and by heat are not consistent. No report has been found which dealt with the effect of light and heat on the color of the different rayons.

Materials

Materials used included three white viscose, three white cellulose acetate, and two white cuprammonium rayon fabrics. The viscose and cellulose acetate rayons each included a flat crêpe, a taffeta, and a satin fabric and the cuprammonium included a fabric of twill weave and a sheer of plain weave. Table 1 gives description and data concerning the physical characteristics of these fabrics.

* Contribution No. 91, Department of Home Economics, Kansas Agricultural Experiment Station.

† Contributions by the junior writer were based on results reported in a master's thesis, "The Effect of Light and of Heat on the Breaking Strength and Color Changes of Viscose and Cellulose Acetate Rayon Fabrics." On file, Library, Kansas State College of Agriculture and Applied Science.

TABLE 1

Description and data of physical characteristics of the fabrics tested.

Fabric	Width (in.)	Luster	Weight per sq. yd. (oz. dry wt.)	% Sizing	Thread count		Crimp %		Yarn counts (deniers, dry weight)		Breaking strength of warp controls (lb.)	
					Warp	Filling	Warp	Filling	Warp	Filling	Dry	Wet
Viscose crêpe	41	Delustered	2.5	0.4	98	72	12.0	6.7	87.2	136.4	32.0±0.1	13.9±0.3
Viscose taffeta	39	High luster	2.8	1.5	111	48	5.6	6.5	126.0	126.0	56.0±0.0	27.3±0.3
Viscose satin	39	High luster	3.4	0.4	135	63	6.4	2.3	84.4	92.6	91.0±1.0	43.4±0.4
Acetate crêpe	38	Delustered	3.7	0.3	59	52	7.0	13.5	249.6	271.8	41.0±0.0	28.2±0.2
Acetate taffeta	39	High luster	2.9	0.6	110	48	6.9	11.0	132.4	127.2	44.5±1.0	24.5±0.6
Acetate satin	39¼	High luster	3.5	1.2	136	66	4.4	6.6	87.8	81.0	52.0±0.0	28.9±0.3
Cuprammonium sheer	39	Delustered	2.0	1.4	121	73	2.5	16.1	76.4	67.0	23.6±0.1	12.0±0.2
Cuprammonium twill	42	High luster	2.5	2.4	83	62	3.8	7.1	121.6	124.8	41.3±0.2	20.8±0.1

Methods

All the fabrics were scoured for 20 minutes with a 0.5% neutral soap solution at 180° F. and rinsed in hot distilled water 12 times to remove all finishing materials. Samples, warpwise of the fabric, were prepared by the raveled-strip method of Committee D–13.[1] The rayon strips were exposed to light from a General Electric Sunlight (Type S–1) lamp for periods of 20, 40, 60, 80, 100, and 120 hours. This lamp gave an intensity of ultraviolet light of 4000 foot-candle minutes as compared to 180,000 foot-candle minutes given by the midsummer noonday sunlight[2, 3] and used 12.4 kilowatt hours of energy in 24 hours. Exposures were made in a standard atmosphere having 65% R.H. at 70° F. The raveled strips of fabric were heated in an electric oven at a temperature of 270–280° F. for 10, 20, 30, 40, 50, and 60 hour periods.

Controls and samples damaged by light and heat were broken dry in a Scott automatic power yarn and cloth tester at standard atmospheric conditions, after they had been kept at such conditions until moisture equilibrium was reached. Strips were broken wet, following exposure, after they had been immersed in distilled water for two hours. Ten wet samples and an equal number of dry samples were broken for each exposure to determine breaking strength and elongation.

The broken samples from the dry breaking strength determinations were analyzed by the method described by the Bausch and Lomb Optical Company[4] for color attributes according to the Munsell system. After the sector percentages of neutrals and colors were determined experimentally, the color specifications of hue, saturation (chroma), and brilliance (lightness or value) were calculated according to formulas in the catalogue.[4] Then these color specifications were reduced to one figure, the index of fading, by Nickerson's formula.[5] In this formula, $I = (C/5)d2H + 6dL + 3dC$, I = index of fading, H = hue, L = lightness (brilliance or value), C = chroma (saturation), and d = difference.

Determinations were made in duplicate of copper number, ash alkalinity, and per cent solubility in sodium hydroxide on the controls and samples for all exposures in order to obtain further evidence of damage to the samples by light and heat.[6]

The copper number, the number of grams of copper reduced from the cupric to the cuprous state in an alkaline solution by 100 grams of dry sample, was determined for all samples by Braidy's method.[7]

The determinations of ash alkalinity[8] were made for a measure of degradation after the samples had been given a standard acid wash. The acid wash removes the alkaline ash from cellulose but leaves it combined with the carboxyl group where oxycellulose had been formed. The samples were steeped in N/10 sulphuric acid, washed 12 times in distilled water, steeped in water for 3 hours, rewashed, and air dried. Approximately 5-gram samples were dried, weighed and ashed in crucibles. The ash was treated with 10 ml. of N/100 sulphuric acid and warmed for 1 hour. The contents were transferred to a flask and diluted with 60 ml. of distilled water and titrated with N/100 sodium hydroxide. A blank was run on 10 ml. of the N/100 acid and the results were calculated in terms of the number of ml. of normal acid per 100 grams of dry sample.

When a = weight of dry sample, b = volume of alkali used for blank,

c = volume of alkali used in test, n = normality of the standard alkali, then ash alkalinity $= \frac{(b - c)n}{a} \times 100$.

The solubility in sodium hydroxide was determined for the controls and all exposures.[8] Approximately 2-gram samples were dried, weighed, and boiled for 1 hour in 200 ml. of N/4 sodium hydroxide (1% solution) in a conical flask with a reflux condenser. After the sample was washed and dried the per cent loss of weight was calculated.

Results

The effects of light and heat on the dry and wet breaking strength and elongation of all the rayons are shown in Figs. 1, 2, 3 and 4. Light decreased the breaking strength of all the rayons, but no one kind showed a marked deviation from the others. Fig. 1 shows the effect of light on

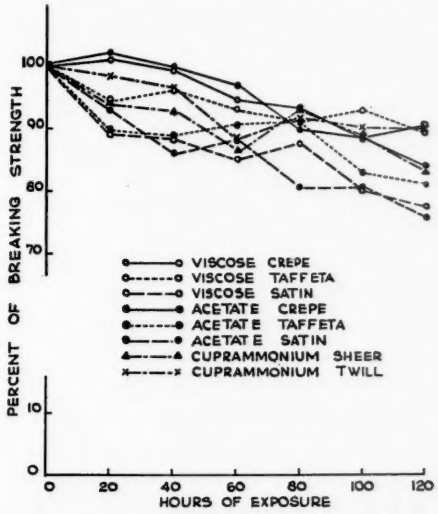


Fig. 1. Dry Breaking Strength in Per cent of Dry Breaking of the Control for Samples Damaged by Light.

the dry breaking strength, and similar results were found for the effect of light on the wet breaking strength. Heat decreased the breaking strength much more than light. Fig. 3 shows the effect of heat on the wet breaking strength of the rayons. Similar results were obtained for dry breaking strength except that the curves were not so steep, and the range for the 60-hour exposure was 36 to 69% instead of from 16.5 to 51% as in the case of the wet breaking strength. In each case the breaking strength of the cellulose acetate rayons was greater than that of the

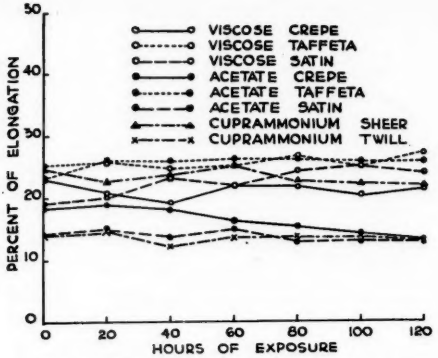


Fig. 2. The Per cent of Elongation of Dry Breaking Strength of Samples Exposed to Light.

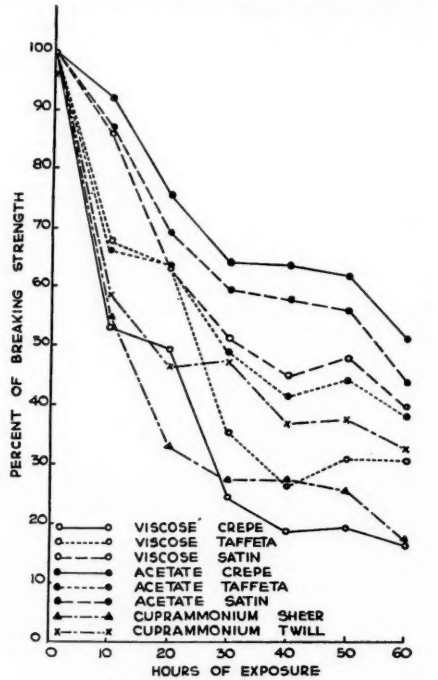


Fig. 3. Wet Breaking Strength in Per cent of Wet Breaking Strength of the Control for Samples Damaged by Heat.

viscose and cuprammonium rayons. Light showed little change on the per cent of elongation of the breaking strength, but heat decreased the per cent of elongation. Fig. 2 shows the effect of light on the elongation of dry breaking strength, and Fig. 4 the effect of heat on the elongation of wet breaking strength. Similar results were found for the effect of light on the elongation of wet breaking strength and the effect of heat on the elongation of dry breaking strength.

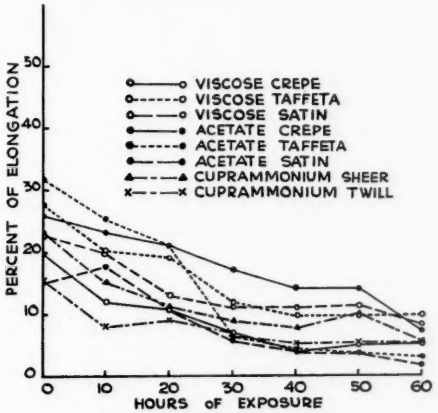


Fig. 4. The Per cent of Elongation of Wet Breaking Strength of Samples Exposed to Heat.

In all fabrics there was little change of color with exposure to light. There was a decided change in color of all fabrics exposed to heat and the changes have been plotted in Fig. 5. The cellulose acetate rayons were affected the least.

The copper numbers for the various exposures of the rayons are shown in Table 2. Light had little effect on the copper number. Heat increased the copper number of the viscose and cuprammonium rayons considerably, but not that of cellulose acetate rayon. The copper number of the controls of viscose and cuprammonium rayons is approximately 1 and that of cellulose acetate rayon is 3.

The ash alkalinities of the rayons exposed to heat are given in Table 3. The results for the samples exposed to light were practically the same as for the heat exposures, and the longer exposures showed no perceptible increase or decrease.

The results of per cent of loss of weight of the fabrics exposed to heat when boiled for one hour in N/4 sodium hydroxide are given in Table 4. The solubility of the viscose and cuprammonium rayons exposed to heat increased from about 5% to 15%, but light had little effect on the loss of weight. The loss of weight of the cellulose acetate rayons increased from 42% to 48%, and the per cent solubility of all samples exposed to light was the same as for the controls.

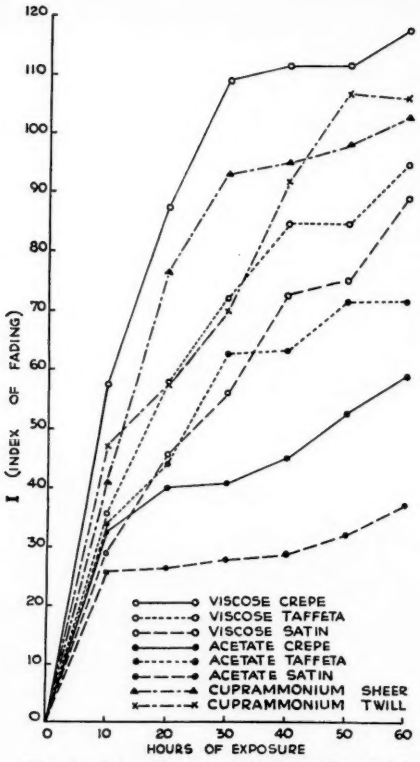


Fig. 5. Color Changes of Samples Exposed to Heat Plotting the I of Nickerson's Formula.

TABLE 2

Copper numbers of viscose, cellulose acetate, and cuprammonium rayon fabrics exposed to heat.

Hours of heat	Viscose crêpe	Viscose taffeta	Viscose satin	Acetate crêpe	Acetate taffeta	Acetate satin	Cuprammonium sheer	Cuprammonium twill
0	1.22	1.13	1.29	3.32	3.11	3.27	1.16	1.00
10	2.61	3.21	2.19	3.35	4.14	3.37	2.27	2.18
20	3.53	3.89	3.19	3.03	4.35	4.32	3.54	2.82
30	4.54	4.20	3.74	3.79	4.59	4.51	4.34	3.26
40	4.80	5.01	3.95	3.43	5.03	4.56	4.65	4.31
50	5.06	5.68	4.09	3.83	5.14	4.79	4.60	4.64
60	5.16	5.52	4.19	3.35	5.20	4.34	5.07	5.13

TABLE 3

Ash alkalinities of viscose, cellulose acetate, and cuprammonium rayon fabrics exposed to heat.

Hours of heat	Viscose crêpe	Viscose taffeta	Viscose satin	Acetate crêpe	Acetate taffeta	Acetate satin	Cuprammonium sheer	Cuprammonium twill
0	1.06	.37	.45	.40	.87	.43	.60	.69
10	1.13	1.06	.97	.47	1.12	.50	1.38	1.65
20	1.04	.64	.74	.39	1.02	.51	1.15	.89
30	1.10	.57	.72	.46	.99	.49	.85	1.03
40	1.08	.61	.72	.41	1.14	.43	.50	.93
50	1.18	.72	.56	.55	1.21	.47	.56	1.23
60	1.19	.80	.47	.46	1.08	.37	.79	1.25

TABLE 4

Percent loss of weight of viscose, cellulose acetate, and cuprammonium rayon fabrics exposed to heat when boiled in N/4 solution of NaOH for one hour.

Hours of heat	Viscose crêpe	Viscose taffeta	Viscose satin	Acetate crêpe	Acetate taffeta	Acetate satin	Cuprammonium sheer	Cuprammonium twill
0	5.5	4.6	4.6	42.3	43.5	43.4	4.8	4.0
10	9.9	8.6	7.4	43.4	44.8	44.1	7.5	7.0
20	10.6	9.1	9.3	44.8	45.0	43.6	10.4	8.6
30	13.7	12.6	10.4	45.7	46.6	45.3	12.2	10.5
40	16.1	14.2	12.7	45.4	48.5	45.4	12.4	13.8
50	17.5	15.5	12.5	46.3	49.2	46.5	13.3	15.6
60	18.3	15.8	12.8	46.5	49.1	48.5	14.0	16.2

References

1. Committee D–13, A. S. T. M. Standards on Textile Materials. Philadelphia. Am. Soc. for Testing Mtls. Oct. 1939, P. 324.

2. Porter, L. C., Egeler, C. E. and Sturrick, W. Ultraviolet Equipments and Their Applications. Cleveland. General Electric Co. Dec. 1931, P. 43.

3. Taylor, A. H. Ultraviolet Radiation from the Sunlight (type S–1) Lamp. *J. Amer. Optical Soc., 21,* P. 20–29 (1931).

4. The Bausch and Lomb HSB Color Analyzer. Rochester, N. Y. Cat. No. 33, P. 14–20.

5. Nickerson, Dorothy. The Specification of Color Tolerances. *Text. Rsch. 6,* P. 505–14 (1936).

6. Marsh, F. J. and Wood, F. C. An Introduction to the Chemistry of Cellulose. D. Van Nostrand Co., Inc. New York. P. 431 (1939).

7. Heyes, T. F. A Micro-method for the Determination of the Copper Number of Cellulose. *J. Soc. Chem. Industry, 47,* P. 90–92 T (1928).

8. Skinkle, John H. Textile Testing. Howes Publishing Co. New York. P. 267 (1940).

Controls for the Size Box

By WILLIAM E. YELLAND *

A Research of U. S. Institute for Textile Research, Inc.

Summary

(1) Closer automatic control of size box temperature is obtained when the size level is also controlled automatically.

(2) In small shallow size boxes, such as exist on certain rayon slashers, the size level can be controlled more effectively by an electrical contact rather than by the use of devices depending on size temperature.

(3) When level or temperature controls are installed in a size box the surface of the control exposed to the size should be composed of a metal as nearly like that of the lining of the size box as possible; otherwise, corrosion—due to electrolytic action set up by dissimilar metals in close contact with each other and immersed in the hot size liquor—may cause serious difficulties.

Temperature and Level Controls for the Size Liquor

The desirability of uniform size temperature has been pointed out in a previous article (*Text. Rsch.*, June, 1940). As soon as they were available, controls were installed in the size box. The temperature control was a motorized valve on the steam line to the size box. It was motivated by a separate unit in the control panel which contained the regain controls (see *Text. Rsch.*, Sept. 1940). The detecting bulb was of the mercury-filled type common in industry. It was mounted close to the bottom and near the middle of the size box so it was always immersed in the size liquor. It also operated a recording unit so a complete record of the size temperature was always available.

A size level control was also installed. It comprised a detecting unit, separate control unit in the large control panel and a motorized valve on the size line. The first detecting unit was a mercury-filled bulb similar to that on the temperature control, mounted in the size box just at the size level. When the size level dropped below the bulb the bulb was supposed to cool off. This drop in temperature would set the mechanism in operation to open the valve on the size line. When the size level rose again the hot size would strike the bulb and the control would then shut off the size.

This method was not satisfactory due to the size and shape of the size box. The box was so narrow and shallow that the bulb could not be mounted sufficiently far enough away from the side of the box on one hand and the

* Dr. Yelland was director at Massachusetts Institute of Technology of the filament viscose rayon phase of the warp sizing research from Jan. 1, 1936, to Sept. 30, 1938, and is now with Corn Products Refining Co., Research Laboratory, Edgewater, N. J.

quetsch roll on the other to allow the bulb to cool off quickly when the size level fell. Enough heat radiated from the side of the box and the roll to keep the bulb hot until the size level had fallen much too low. Consequently the size level was always falling too low, regardless of the position of the bulb, and there was more variation in size level with this type of automatic control than with manual control.

The bulb detecting unit was removed and replaced by a unit in which the size completed a contact between two electrodes. As soon as the size level fell the contact was broken, thereupon the control opened the valve

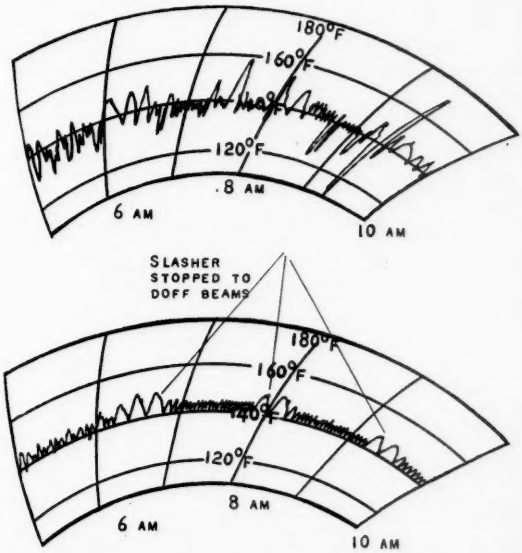


Fig. 1.—Sections of Chart Records of Size Temperature. Top Section—Both Controls Automatic. Bottom Section—Automatic Temperature Control and Manual Level Control.

in the size line and more size entered. When the level had risen to complete the contact the size line was again closed. This device was independent of the temperature and required only that the liquid be capable of conducting a very slight electric current. It did not work with pure water but functioned well with different types of size mixes. It tended to give difficulty only when the size foamed badly as the foam layer acted in the same way as the size liquid itself.

If the slasher tender were careless and touched the electrodes while the current was on he might receive a slight shock. However, it was not dangerous and the men learned very quickly to leave the electrodes alone when the machine was in operation.

With a unit of this type it was possible to control the size level very closely. Better control of the size level afforded better control of the size temperature. (See Fig. 1 for sections of chart records.)

Automatic level control necessitated screens in the size lines in front of the motorized valve. These screens must be able to be removed quickly and readily for cleaning, otherwise considerable difficulty will be met with by extraneous material clogging the motorized valve.

Corrosion in the Size Box

Two dissimilar metals in close contact with each other, and with a liquid which is capable of conducting an electric current, form an electrolytic cell which leads to the corrosion of the metal higher in the electromotive series. This situation is occasionally encountered when control units are being installed in the size box.

When the temperature control was first installed the thermometer bulb was stainless steel. It was clamped to the inside of the size box with copper brackets. After a month of service the control failed to function properly. Examination of the bulb showed that corrosion had occurred in the stainless steel of the bulb immediately adjacent to and under the copper brackets to such an extent that the mercury in the bulb was allowed to leak out.

The size mix was not corrosive as measurements showed that it had a pH 6 to pH 7. (Pure water has a pH 7.) Moreover, the main body of the bulb which had also been immersed in the size, but which had not been in contact with the copper bracket, was not even tarnished. The bulb was replaced by one plated with bronze. This bulb after having been in operation under the same conditions as the other for over three months showed no signs of corrosion.

Reaction of Silk Fibroin with Diazomethane

By HENRY A. RUTHERFORD, WILBUR I. PATTERSON and MILTON HARRIS *

A Textile Foundation Publication

Abstract

The reactive groups in silk are intimately related to the absorption of moisture, acids, alkalies, or dyes, and to physical properties such as extensibility and tensile strength. A large proportion of these gorups are present in silk fibroin in the form of hydroxyl groups of the amino acid tyrosine. The latter groups are of special interest, partly because of their preponderance, but principally because there are divergent views as to whether they are free in the untreated fibres. As an approach to the problem it appeared advisable to attempt to inactivate these groups by suitable chemical treatment and then to study the properties of the modified protein. Several methods have been tried for this purpose by different investigators but they were found to be too drastic, and generally resulted in severe physical deterioration of the fibre.

In the present investigation, the silk was treated with an ethereal solution of the methylating agent diazomethane. The treatment resulted in a rapid decrease in tyrosine content of the silk with no appreciable decrease in tensile strength of the fibres, and the results led to the conclusion that the hydroxyl groups of tyrosine are free in the untreated fibres. The total methoxyl content after treatment of the fibre for 25 hours or longer was greater than could be accounted for by assuming that only phenolic hydroxyl groups had reacted. The "extra" methoxyl was not on either the primary amino or the serine hydroxyl groups, but appears to have resulted from the reaction of diazomethane with groups, tentatively assumed to be carboxyl, to form the methyl esters.

Introduction

MANY of the chemical and physical properties of a protein are intimately related to the number and arrangement of its functional or reactive groups, most of which are contributed by the dibasic and diacidic amino acids. Silk fibroin is composed largely of the amino acids, glycine, alanine and tyrosine, and of these only the last one would be expected to contribute a reactive group, namely, a phenolic hydroxyl group. This group thus becomes of special interest.

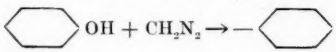
* Research Associates at the National Bureau of Standards representing the Textile Foundation, Inc.

As the result of observations in this laboratory, it appeared that the hydroxyl groups of tyrosine might not be free in the untreated fibre. For example, it was found that when fibroin was treated at pH 8 with Folin's phosphomalybdate phenol reagent,[1] only a pale blue color, indicative of phenolic hydroxyl groups, was produced, but as the pH was increased the solution became increasingly colored. Pretreatment of the silk with alkali also increased the depth of color obtained with this reagent. Similar results were obtained with other proteins (pepsin and wool) and it appeared that not all of the phenolic hydroxyl groups of these proteins were free. Further evidence favoring this view was obtained by Fruton and Lavin [2] who observed that the characteristic absorption bands of tyrosine were not found in untreated papain, although on acid hydrolysis appreciable amounts of the amino acid were liberated.

The state of this hydroxyl group in the silk fibre becomes of considerable importance, since it would be expected to influence such properties of the fibre as tensile strength, extensibility, and absorption of moisture, acids, alkalies or dyes. Unfortunately, the experiments indicating that the hydroxyl groups are not free must be considered inconclusive, and open to some criticism since they involve the use of treatments which are known to have a degradative effect on the protein. It appeared that a better approach to the problem might be made by attempting to cause the hydroxyl group to react with suitable mild chemical reagents under conditions which would minimize the possibility of degradation of the fibres.

Of the available reagents, the acid chlorides or anhydrides, ketene, and diazomethane appear to be most suitable for inactivating the hydroxyl groups of tyrosine. The acid chlorides and anhydrides were used for this purpose by Abderhalden and Brockmann,[3, 4] but were found to produce appreciable deterioration of the fibres. Ketene was used by Kise and Carr [5, 6] with far more satisfactory results, although on prolonged treatment with this reagent, the fibre was colored tan and some degradation occurred. The product of the reaction of ketene with the hydroxyl group of tyrosine is an ester which is readily hydrolyzed in either alkaline or acid solutions. For this reason, it is not possible, by the usual tyrosine analyses, to estimate quantitatively the extent to which the ketene has reacted with the hydroxyl groups. Further, the acetyl value of the treated fibre is also inadequate for this purpose since it is known that the ketene reacts with groups other than the phenolic hydroxyl group.

Diazomethane offers several advantages over the above-mentioned reagents. It can be used in neutral aqueous or organic solutions and it reacts with a phenolic hydroxyl group to form an ether as follows:



$$\text{OH} + CH_2N_2 \rightarrow \text{OCH}_3 + N_2 \qquad (1)$$

The product of the reaction is stable during the alkaline hydrolysis used in tyrosine determinations, which makes possible the quantitative estimation of the extent to which the tyrosine has reacted. The reagent was used by Herzig and Landsteiner,[7] and by Abderhalden and Brockmann [3] on several proteins including silk but the extent of the reaction with tyrosine was not determined. The methoxyl contents of the treated fibres, however, were found to be greater than would be expected if only the phenolic hydroxyl groups were reacting.

Experimental Procedure

1. Materials and Methods.—Silk fibroin in the form of a plain-woven cloth was used in this work. Before treatment with diazomethane it was extracted for 8 hours each with alcohol and with ether, and finally washed thoroughly with cold distilled water.

Total nitrogen was determined by the micro-Kjeldahl method, tyrosine by Lugg's method[8] after an alkaline hydrolysis, amino nitrogen by the method of Rutherford, Harris, and Smith,[9] methoxyl content by the procedure described by Vieböck and Brecher,[10] and methyl alcohol by Beyer's method.[11]

2. Preparation of Diazomethane.—The ethereal solution of diazomethane was prepared according to the method described by Adamson and Kenner.[12] The apparatus is shown in Fig. 1. The temperature of the reaction flask

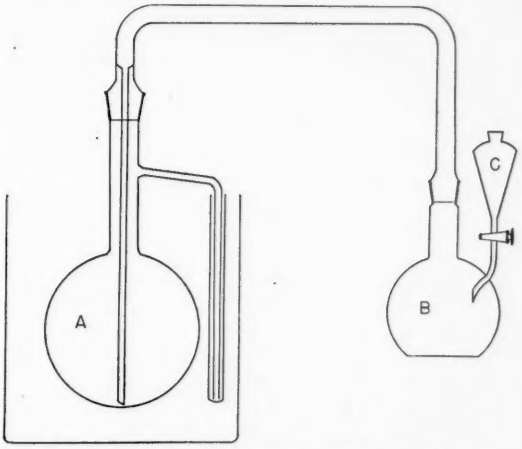


Fig. 1. Apparatus for preparation of diazomethane.

B was maintained at 75° to 80° C in a water bath, and that of the receiver A at —10° to —15° in a brine tank. Before the distillation of the diazomethane was started, about 100 ml of ether was added to flask A, and 10 ml to the trap at the side arm of A. 0.05-mole of the nitroso derivative of the methylamine addition product of mesityl oxide in 40 ml of ether and 6 ml of isopropanol was added dropwise from funnel C to 30 ml of 1% sodium isopropoxide in flask B, the rate of addition being slightly greater than that of distillation. When the addition was complete, 15 ml more of 1% sodium isopropoxide was added to B, followed by another 0.05-mole portion of the nitroso ketone as before. Finally, ether was added to flask B until all the diazomethane was distilled into A. By this procedure about 1 g of diazomethane in 250–300 ml of ether was obtained. The actual amount was readily determined by the addition of an aliquot of the ether solution to a weighed quantity of benzoic acid, and subsequent titration of the excess acid with 0.1 N potassium hydroxide in alcohol.[13]

3. Methylation of Fibroin.—Preliminary experiments showed that the rate of methylation of the tyrosine hydroxyl groups of the silk was increased by the presence of a little moisture, by increased concentrations of diazomethane, or by treating the fibres at low temperature (— 10° C or below). On the basis of these experiments the following procedure was used: The sample was first wet thoroughly with 75% alcohol and then squeezed as dry as possible. This wetting served to introduce an appreciable quantity of water into the fibre, and when the sample was treated by this procedure, the subsequent separation of a water layer in the ether solution was avoided. The silk was then placed in flask A containing the diazomethane, and the reaction allowed to proceed at — 10° to — 15° C, for the desired length of time. A rapid rate of reaction was obtained when the ratio of diazomethane to silk was about 1: 5. This is 4 to 5 times the amount required to completely methylate the fibroin. This concentration may be maintained approximately by distilling more diazomethane into flask A as the reaction proceeds. After the treatment, the silk was removed and the excess ether and diazomethane were allowed to evaporate. The samples were thoroughly washed in successive portions of distilled water to remove the remaining organic solvents and then allowed to dry.

Results and Discussion

1. The Reaction of Silk With Diazomethane.—The extent of methylation of the tyrosine hydroxyl groups of the silk was determined by analysis of the treated material for tyrosine. The results in Table 1 show the

TABLE 1

Tyrosine and methoxyl contents of silk fibroin after treatment for different lengths of time with an ethereal solution of diazomethane.

Duration of Treatment	Tyrosine content	Methoxyl content of		
		Fibroin	Hydrolysate	Hydrolysate calc. from decrease in tyrosine content
hrs.	%	%	%	%
0	12.1	—	—	—
1	6.6	0.92	0.94	0.94
1.5	4.1	1.94	1.49	1.37
3	3.6	1.97	1.54	1.45
5	3.2	1.99	1.52	1.52
10	2.4	2.32	1.60	1.66
20	2.3	2.35	1.58	1.68
35	2.0	2.55	1.76	1.73
50	1.6	2.62	1.86	1.80

tyrosine contents of samples treated for different lengths of time. The rate of reaction is quite rapid at first and gradually decreases as the treatment is continued. In 10 hours, 80% of the total tyrosine is converted into the methyl derivative. Treatments for longer periods of time, during which

the concentration of diazomethane was kept nearly constant, resulted in a slight further decrease in tyrosine content. The rate of reaction may in part depend on penetration of the reagent into the fibre, and it might accordingly be expected that those groups on and near the surface would be methylated most rapidly. No appreciable decrease in tensile strength of the fibres occurred even on prolonged treatment. Since practically all of the tyrosine in silk is so readily methylated under these conditions, it appears that the hydroxyl groups of this amino acid are free in the untreated fibre.* A possible explanation of the discrepancy between this conclusion and that of the experiments with Folin's phenol reagent previously described might be found in a consideration of the nature of the reaction in the Folin method. This reaction depends on the oxidation of a phenolic hydroxyl group by the reagent, the latter in turn being reduced to the highly colored state. The depth of color developed by a known amount of tyrosine is used as a standard. If, however, the oxidation potential of the hydroxyl group of the tyrosine as it exists in the protein is different from that of the tyrosine itself, then the apparent tyrosine values obtained for the protein will not be true values. On this assumption, it would appear that the oxidation potential of the hydroxyl group is greater when the tyrosine is part of the protein molecule.

An examination of the data in columns 3 and 5 of Table 1 shows that the methoxyl content of the treated silk is greater than can be accounted for if it is assumed that only the hydroxyl groups of the tyrosine had reacted. Presumably, the "extra" methoxyl results from the reaction of diazomethane with other groups in the fibre. Conceivably, diazomethane might react with silk in several ways, as shown in the following equations:

$$\text{P–CH}_2\text{–C}_6\text{H}_4\text{OH} + \text{CH}_2\text{N}_2 \rightarrow \text{P–CH}_2\text{C}_6\text{H}_4\text{OCH}_3 + \text{N}_2 \qquad (1)$$

$$\text{P–CH}_2\text{NH}_2 + \text{CH}_2\text{N}_2 \rightarrow \text{P–CH}_2\text{NHCH}_3 + \text{N}_2 \qquad (2)$$

$$\text{P–CH}_2\text{COOH} + \text{CH}_2\text{N}_2 \rightarrow \text{P–CH}_2\text{COOCH}_3 + \text{N}_2 \qquad (3)$$

$$\text{P–CH}_2\text{OH} + \text{CH}_2\text{N}_2 \rightarrow \text{P–CH}_2\text{OCH}_3 + \text{N}_2 \qquad (4)$$

where, P represents the protein molecule to which the above groups are attached. The equations then represent the reaction of diazomethane with (*1*) the hydroxyl groups of tyrosine, (*2*) the free amino groups, (*3*) the free carboxyl groups, and (*4*) the hydroxyl groups of serine.

Reaction (*1*) is shown to occur by the decrease in tyrosine content of the treated silk. Reaction (*2*) would result in the formation of a methylamino compound which would not give methoxyl under the analytical conditions used, and would therefore not account for any "extra" methoxyl. Moreover, amino-nitrogen determinations as shown in Table 2, indicate that this reaction does not occur. Reaction (*3*) undoubtedly occurs since diazomethane is known to readily methylate carboxylic acids with the formulation of methyl esters. Upon hydrolysis of the fibres under the conditions generally used in the tyrosine determination (6 *N* NaOH for 20 hours at 105° C) such esters would be expected to decompose liberating methyl alcohol with the regeneration of the carboxylic acid. The former would be evolved, and thus the methoxyl content of the hydrolysate should represent

* It is of interest to note that the tyrosine contents of silk sericin, crystalline pepsin, and wool were reduced from 6.3, 7.8 and 5.8% to 0.5, 1.8, and 1.4% respectively after treatment for 20 hours with diazomethane.

TABLE 2

Total and amino-nitrogen contents of silk after treatment for different lengths of time with diazomethane.

Duration of treatment	Total N	Amino N
hrs.	%	%
0 (untreated)	18.78	0.10
20	18.82*	0.10
50	18.81*	0.10

* Corrected for increase in weight due to the addition of methyl groups.

those methoxyl groups which resist the hydrolytic conditions used in these experiments. Reactions (*1*) and (*4*) would give this type of methoxyl.

The methoxyl contents of the hydrolysates are shown in the fourth column of Table 1. As was expected, the values are lower than those obtained for the treated silk (column 3). The agreement of the hydrolysate values with the methoxyl values calculated from the decrease in tyrosine contents (column 5), however, leaves little likelihood that reaction (*4*) occurs to any appreciable extent, especially since complete methylation of the serine of silk (1.8%) [12] would yield 0.53% of methoxyl groups or about 30% of the total found.

The liberation of methyl alcohol from methylated silk during hydrolysis was readily demonstrated by refluxing several different samples for 1 hour with 0.1 *N* NaOH, and then distilling about half the solution into a receiver maintained at 0° C (to avoid loss of CH_3OH). The methyl alcohol content of the distillate was determined colorimetrically by Beyer's method. The results, calculated as methoxyl, are shown in Table 3.

TABLE 3

The effect of a 1 hour treatment with a boiling 0.1 *N* solution of sodium hydroxide on the methoxyl content of methylated silk.

Duration of diazomethane treatment	Total methoxyl content	Methoxyl liberated during hydrolysis as methyl alcohol	Residual methoxyl content of silk
hrs.	%	%	%
3	1.97	0.30	1.55
10	2.32	0.50	1.85
50	2.62	0.73	2.08

If it is tentatively assumed that the "extra" methoxyl in the treated silk is in the form of esterified carboxyl groups, the present results indicate that silk fibroin contains an appreciable quantity of dicarboxylic amino acids, though this seems not to have been reported in the literature. From the data in Table 1, it is estimated on the basis of the above assumption, that silk may contain as much as 0.26 millimole per gram of free acidic groups. Such an appreciable amount of acidic groups would be

expected to considerably influence the acidic and basic properties of the silk, and should be readily detectable in the titration curve of the fibre.*

During these studies it was observed that an appreciable quantity of alkali was liberated when the fibroin was washed with water subsequent to the diazomethane treatment. A possible explanation for this is that cations, held by the acidic groups of the fibre, are freed as these groups react with diazomethane. Since the reaction was carried out in ethereal solution, the alkali was not removed from the fibres until they were washed with water. If such an explanation is correct, no alkali should be liberated from ash-free silk under similar conditions. Furthermore, the rate of reaction of ash-free silk with diazomethane might be very different than that of the untreated silk since at least a portion of the acidic groups of the latter would already be combined with inorganic substances.

In order to test this explanation and its consequences, a sample of ash-free fibroin was prepared by electrodialysis and then treated with diazomethane for different lengths of time. It was found that no measurable quantity of alkali was liberated when the treated sample was washed with water. Furthermore, the rate of increase in methoxyl content of the electrodialyzed silk during the first hour of treatment was greater than that of the untreated fibres as shown by the results plotted in Fig. 2. After

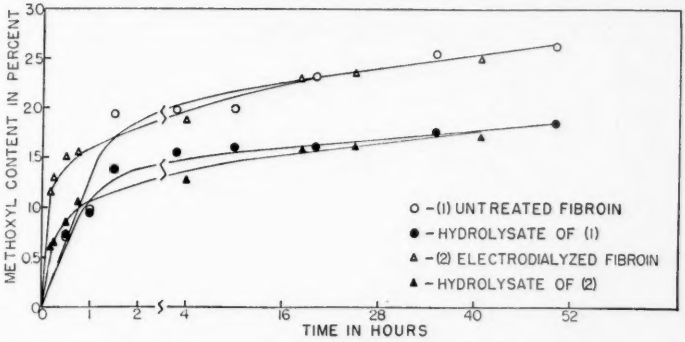


Fig. 2. Rate of reaction of untreated and electrodialyzed fibroin with diazomethane.

one hour of treatment no "extra" methoxyl was found in the latter while treatment of the electrodialyzed fibres for the same length of time resulted in the formation of about 65% of the maximum "extra" methoxyl. After about 20 hours, there was no further increase in "extra" methoxyl content. As in the case of the untreated silk, the difference between the total and hydrolysate methoxyl contents of the electrodialyzed sample becomes approximately constant and equal to about 0.26 millimole per gram.

Although no further work on this phase of the investigation has been done, it appears from the rate studies that the specificity of diazomethane

* The titration curve of silk is now being determined as part of a separate investigation in progress in this laboratory.

for the hydroxyl groups of tyrosine might be increased by appropriate pretreatment of the protein with some inorganic substance for the purpose of blocking the carboxyl groups.

2. Reaction of Methylated Silk With Nitrous Acid.—The methylated and untreated samples of silk were found to behave quite differently toward nitrous acid. The untreated silk assumed the usual dark brown color, while the depth of color formed in the diazomethane-treated sample appeared to be dependent on the extent of methylation. A sample in which the tyrosine content had been reduced to a very low value was colored only slightly by nitrous acid.

Philpot and Small [14] have suggested that the color resulting from the reaction of pepsin with nitrous acid may be due to a nitrosation of the tyrosine, and that the first product of the reaction is an ortho-nitroso derivative. Assuming a similar reaction for silk, it appears that the methylation of the tyrosine prevents the formation of this derivative, either by decreasing the reactivity of the ortho position, or by preventing reaction of the nitrous acid with the free hydroxyl group to form a compound which subsequently rearranges to the ortho position. Regardless of which of the above mechanisms prevails, the difference in the behavior of methylated and untreated silks toward nitrous acid may be considered additional evidence that hydroxyl groups of the tyrosine in untreated silk are free.

References

1. Folin and Ciocalteu. *J. Biol. Chem., 73,* 627 (1927).
2. Fruton and Lavin. *J. Biol. Chem., 130,* 375 (1939).
3. Abderhalden and Brockmann. *Biochem. Z., 226,* 209 (1930).
4. Abderhalden and Brockmann. *Biochem. Z., 211,* 395 (1929).
5. Kise and Carr. *Text. Rsch., 7,* 103 (1936).
6. Carr. *Text. Rsch., 8,* 125 (1938).
7. Herzig and Landsteiner. *Biochem. Z., 61,* 458 (1914).
8. Lugg. *Biochem. J., 31,* 1422 (1937); *32,* 775 (1938).
9. Rutherford, Harris, and Smith. *J. Rsch. NBS., 19,* 467 (1937) RP1038.
10. Vieböck and Brecker. *Ber. deut. chem. Ges., 63B,* 3207 (1930).
11. Beyer. *J. Assoc. Official Agr. Chem., 22,* 151 (1939).
12. Adamson and Kenner. *J. Chem. Soc.,* 286 (1935).
13. Abderhalden. *Z. Physiol. Chem., 120,* 207 (1922).
14. Philpot and Small. *Biochem. J., 32,* 534, 542 (1938).
15. Marshall and Acree. *Ber. deut. chem. Ges., 43,* 2324 (1910).

Factors Influencing Flocculation and Precipitation. Theory and Variables*

By E. M. BEAVERS, J. E. MAGOFFIN and F. K. CAMERON

A Textile Foundation Publication

Flocculation and precipitation of a colloidal dispersion are inseparably related in as much as the phenomena associated with the latter process are closely dependent upon those of the first. This paper will offer a brief discussion of the information relevant to both these processes, and of the variables to be encountered in such an investigation. Reports to follow will present the results of experimental work.

Theory of Sedimentation

STOKES,[37] in 1850, derived the law describing the rate of settling of individual particles under conditions of "viscous flow." If the particle be spherical, then it can be shown that

$$v = \frac{2r^2(d_p - d_m)g}{9\eta} \qquad (1)$$

where v is the velocity of settling, r and d_p are the radius and density of the particle, respectively, d_m is the density of the medium, g the acceleration of gravity, and η the coefficient of viscosity of the medium. If the particle be not spherical, the expression may be written

$$v = K\frac{A(d_p - d_m)g}{\eta} \qquad (2)$$

where K is a constant dependent upon the shape of the particle, and A is the particle's surface area.

Further conditions imposed upon these equations are (1) that the medium be limitless in extent; i.e., that there be no wall-particle or particle-particle interaction effects; (2) that the particle be rigid; (3) that no shear occur at the interface between particle and medium, and (4) that the particle be large in comparison to the size of the molecules of the medium.

Modifications [11] have been made to Stokes' law to allow for departures from the above conditions. If settling be so rapid that some of the particle's energy be used in setting up eddy currents, then viscous flow no longer obtains. Under conditions of eddying resistance, the velocity is directly proportional to the square root of the particle radius: [2]

$$v = k\sqrt{\frac{r(d_p - d_m)}{d_m}} \qquad (3)$$

where k is a constant.

* This investigation was made under the auspices of the Textile Foundation, and under the direction of Dr. F. K. Cameron at the University of North Carolina.

Faxen[15] has corrected Stokes' law for the container "wall effect" by introducing a term involving the ratio of particle diameter to tube diameter. The correction becomes negligible for very small particles and for settling vessels of large diameter. Other modifications[7] include corrections for the height of the column of suspension.

An equation intended to include the effect of one uncharged particle on another has been proposed by Kermack, M'Kendrick and Ponder:[23]

$$v = v_0(1 - k'\phi) \tag{4}$$

where v_0 is the settling velocity for a single particle as calculated by (*1*), ϕ is the average volume of suspended material per unit volume of suspension, and k' is a constant.

The correction of Stokes' law for such surface effects as slip at the interface has been made by Millikan.[26] The existence of any effect of this sort would tend to produce a velocity greater than that calculated from the simple form of the law. The formula, if the effects are small, is:

$$v = \frac{2r^2(d_p - d_m)g}{9\eta}\left(1 + A\frac{b}{r}\right) \tag{5}$$

in which b is a constant of the medium, r the radius of the particle, and A is a constant.

A case of sedimentation in which the medium is "infinite" in extent (i.e., where the falling particle never reaches an end point) would, of course, hold little interest for practical investigations, since in this application it is desired that the suspended material accumulate at the bottom of its container as quickly and as closely as possible. "Free" settling (that which obeys Stokes' law) is not realized in such a situation and for suspensions of lyophobic powders, Robinson[30] has proposed the relation:

$$v = \frac{dH}{dt} = \frac{kr^2(d_p - d_s)}{Z} \tag{6}$$

where H is the height of the suspension, t the time of settling, and d_s and Z the density and relative viscosity, respectively, of the suspension. The relative viscosity may be evaluated in terms of the relative concentration: knowing the dry bulk density and true density of the particles, the initial height and concentration of the suspension, and the manner in which viscosity varies with the height of the suspension, a graphical integration of equation (*6*) will permit a prediction of the settling curve. This method has been found to be highly accurate for certain cases. It assumes that the particles will ultimately pack from suspension to the same condition as that observed in the dry bulk, and therefore that the particles' size and shape are not affected by dispersion in the medium. The method should not be expected to be applicable to suspensions of solids appreciably altered by contact with the medium.

The fact that truly spherical particles are almost never met in practice has led to the proposal of many elaborate forms[16] of Stokes' law. The difficulty in such attempts is that the particles in any one suspension may have many different forms to which no one equation may be assigned. Consequently, it has become customary to speak of the "equivalent sphere"—the hypothetical sphere which would have the same settling rate as the actual non-sphere. If the equation be written in the form of (*1*), then r is the radius

of the equivalent sphere. Studies by Andreasen [1] on flint particles in water have shown that the values for grain size obtained with Stokes' law and sedimentation data agree well within the limits of experimental error with those obtained photomicrographically. Hence, for flint particles at least, the law holds as well for non-spherical as for spherical particles.

It has been pointed out that particles which settle faster than a certain critical velocity do not obey Stokes' law, but follow a square-root relation, (*3*). Similarly, very small particles which begin to come under the influence of Brownian movement show an ever greater departure from the predicted behavior.[22] The well-substantiated distribution equation tested by Perrin and Nordlund [27] describes this condition.

A difficulty, frequently overlooked in the application of the Stokes equation, is that encountered in evaluating the density and viscosity terms. Use of the dry mass density of the solid is customarily resorted to for the d_p term. That this value may be very far from the true one is evident when the adsorptive properties and possible aggregate form of finely dispersed particles are considered. Williams[40] has mathematically described the effect of adsorption on radius and density of particle.

Furthermore, it is questionable whether changes in the settling rate of a suspension may be entirely attributed to changes of particle size since the conditions necessary to bring about the change will affect, in some degree, all the other variable factors in equation (*1*).

As has been demonstrated by a number of investigators,[38] the existence of a charge on the settling particles causes an increase in the effective viscosity. A formula for the case has been derived by von Smoluchowski,[34] but it involves the zeta potential whose calculation is of doubtful validity, and the nature of which is of doubtful character.

Blom [5] and Piechowicz [29] have suggested that for at least some cases of sedimentation the Stokes equation should include a function of the medium's surface tension and the heat of adsorption. Blom [6] later showed that the sediment volumes of several suspensions are a direct function of the heat of vaporization of the medium. Boutaric and Semelet [8] have demonstrated that lowering the surface tension of As_2S_3 sols with camphor, menthol, etc., has no marked effect on their stability unless coagulants be added.

It becomes apparent from this discussion that in very simple and nearly ideal cases, such as Millikan's oil-drops, or Andreasen's flint particles, settling behavior follows fairly well-defined lines, but in the more involved cases of suspensions as they are daily met in water purification, clarification of trade wastes, etc., our knowledge of the mechanics of the process and its influencing factors is very meager. Progress toward better control in the latter instances can come only through further investigation from a fundamental point of view of lyophilic and complex suspensions.

Flocculation

It has been said of sedimentation that "the real key to the situation, from a practical standpoint, is to build up from individual small particles something larger which will settle more rapidly. Therefore the real problem, the real art of sedimentation, is concerned with the art of floc building." [17]

As initially formed, a floc is usually of a light, feathery nature, containing comparatively large amounts of water which may be chemically bound, adsorbed, or entrained in the mesh of the structure. The "tightness" with

which this water is held, and the compactness of the floc's form will determine to a large extent the rate of settling. There has been very little investigation of the conditions which determine the ultimate physical nature of the clump, although the question is one of paramount importance. Here, more than in any other feature of sedimentation, lies an opportunity for the widest control. According to Stewart and Roberts,[36] "the largest and most difficult field of flocculation lies in water, sewage, and the treatment of trade waste."

Flocculation may be brought about in a number of ways. Hartman [20] lists the following influences: (1) mechanical agitation, (2) temperature, (3) alteration of electrical charges, (4) radiant energy of various wavelengths, (5) sound waves, (6) alteration of extent of solvation of particles. The first, third and last are of most interest in the problems with which we shall be largely concerned.

It has been demonstrated [20, 36] that in certain cases, mechanical agitation may not only hasten the rate of flocculation, but also may result in the floc's being more dense and compact.

Some success has been achieved with "weighting" the flocs to improve their rates of settling. Adding chalk or other loading material to coal and clay slurries before coagulation has been found to increase the density of the floc and its settling rate.[32] Steenhoff and Funke [35] added carbon black to "$Al_2(SO_4)_3$ sols" and observed a decided increase in the speed of sedimentation after flocculation.

The only attempts to generalize the action of electrolytes on fine dispersions were made by Schulze [33] and Hardy; [18] the "Schulze-Hardy Law" qualitatively states that the higher the valence of an ion added to a colloidal system of opposite charge, the greater is its coagulating power, usually increasing very rapidly. Through want of a better rule, this frequently serves as the criterion for selection of precipitating agents, although it has been definitely shown not to be applicable in all cases.[3] Experimental work in this field indicates clearly that the efficiency of an electrolyte in coagulation depends upon the characteristics (adsorbability, solvation, valency, etc.) of both ions, and upon the specific nature of the suspension. While it is generally held that the ions of the greatest adsorptive power are the best coagulating agents, Dhar and coworkers [13, 14] have shown that with many suspensions the exact opposite is true, that the ion which is adsorbed less than another is the better coagulator. What may be the true quantitative statement of a rule like the Schulze-Hardy is an important subject for further investigation.

The extent of hydration of particles stabilized by the medium may be altered by the use of such dehydrating agents as alcohol or acetone, as found by Kruyt and coworkers,[25] by which means a lyophilic colloid may be altered to have the properties of a lyophobic one. Bancroft [3] discusses many examples of the precipitation of colloids by alcohol. It is not necessarily true that all the effects observed are due alone to the alcohol's dehydrating property.

Briggs [9] has developed an excellent theoretical conception of the mechanism of coagulation of lyophobic sols based on the work required to bring two particles together against the force of their electrical repulsion and the interfacial work done in causing adhesion of the particles. These two quantities are:

$$W_e = \int \frac{e^2}{\Delta\epsilon} d\Delta \qquad (7)$$

and

$$W_i = \sigma_i(S_b - S_a) \qquad (8)$$

where W_e and W_i are the electrical and interfacial work quantities, e is the charge per unit area of the particles, Δ is the distance between the centers of repulsion of the particles at the moment they begin to move toward each other, ϵ is the dielectric constant of the medium, σ_i is the solid-liquid interfacial tension, and S_b and S_a are the solid surfaces exposed to the medium before and after coagulation. These relations emphasize the fact that coagulating agents may alter other properties than charge in bringing about the coalescence, and suggest investigations which have not been hitherto successfully pursued.

Variables in a Sedimentation Study

It may well have been concluded from the foregoing discussion that simply deciding on a profitable starting point with the purpose of obtaining useful information about the sedimentation of the more complex systems constitutes a problem in itself. Among the possible independent variables in such a study are (1) the kind of suspension, (2) its concentration, (3) its degree of dispersion, (4) temperature, (5) the kind of electrolyte added, (6) the electrolyte concentration, (7) the "age" of the suspension after addition of the coagulant, (8) the kind and amount of non-electrolyte added, (9) the degree of agitation, and (10) the nature of the vessel in which the study is made. Some of the possible variables which might be considered dependent are surface tension, viscosity, densities, heat of adsorption, dielectric constant and charge. A few of these have already been discussed; others will be given brief treatment in this section.

Defining the kind of suspension at the outset frequently presents great difficulties because the mixture's composition may not be constant from time to time, nor even subject to accurate analysis at any one time. The investigator must then resort to study of a reproducible system whose general properties are similar to the average characteristics of the type of suspension to which the results will be applied in practice.

The concentration of solids in a suspension is usually found to be an instrumental factor in determining the settling rate.[39] Often the more dilute suspension settles faster than the more concentrated because of less interference between particles.[12] If the concentration becomes high enough, there may be no free settling at all, but simply a "squeezing out" of the medium from between the particles by virtue of their own weight. However, in some cases, the most rapidly settling particles may entrap and carry down with them some of the slower ones, resulting in a process analogous to filtration so that the efficiency of separation increases with concentration. Berczeller and Wastl [4] observed that when kaolin settles from a concentrated suspension, the supernatant liquid is clear, whereas settling from more dilute suspensions always leaves a cloudy supernatant liquid. Because of the same type of "filtering" sedimentation, Deerr [12] found that tall settling tanks are preferable to flat ones for the clarification of cane juice suspensions. The fact that cream separates most rapidly in a tall container is a time-honored example of the same phenomenon. In most cases, the capacity of a settling unit (e.g., the Dorr clarifier) is dependent upon the area only of the tank.

Although obviously important, no thorough study has been made of the influence of initial degree of dispersion, or average particle size distribution, on the process of flocculation.

In following the variation of settling velocity with change in concentration of a particular electrolyte, a point of maximum efficiency is usually reached,

after which further additions of the coagulating agent show either no change in the sedimentation rate or a decrease in rate caused by repeptization of the suspension. The advantage in establishing the efficient point in practice is obvious.

Il'in [21] has shown that, with some suspensions, a "hysteresis" of sedimentation is exhibited, depending upon whether the velocity be measured immediately after mixing the suspension and the coagulator or at the end of a time interval after mixing. Oden [28] found that the time required for coagulation of $BaSO_4$ suspensions decreases with increasing concentration of the solid, and with increasing concentration of the coagulant.

The sedimentation velocity of kaolin was found by Rona and György [31] to be markedly increased by non-electrolytes, the effect increasing with the length of the chain in the members of homologous series. The settling rate of charcoal was not affected by non-electrolytes.

Kermack and Williamson [24] have shown that under some conditions the effect of silicic acid is to produce the precipitation of a film of insoluble material on the surface of clay particles, causing abnormally rapid sedimentation. Under other conditions, the acid prevents precipitation. A monomolecular film of oleic acid on the surface of a fine powder, suspended in a very dry, non-polar liquid, was found by Harkins and Gans [19] to deflocculate the powder and to settle it to a fourth the volume it would occupy if no oleic acid were present.

The ideal vessel for making settling rate determinations would have the widest possible diameter commensurate with laboratory facilities and within the bounds of manipulative convenience. In addition to the wall effects mentioned earlier, some suspensions tend to form loosely cohesive bridges across narrow tubes,[10] retarding the settling velocity. Measurements made in a narrow tube with such a suspension would give a completely false prediction of the behavior of the same suspension in settling tanks a hundred or more feet in diameter.

The next paper in this series will present an investigation of the influence of particle size distribution on the flocculation and precipitation of colloidal suspensions. Some of the dependent variables in the process (surface tension, viscosity, density, migration velocity, and adsorption) will be experimentally followed to determine their contributions to the observed behavior.

References

1. Andreasen, *Kolloid-Z.*, *48*, 175 (1929).
2. Badger and McCabe, "Elements of Chemical Engineering," 2nd Ed., P. 574 (1936).
3. Cf. Bancroft, "Applied Colloid Chemistry" (1932).
4. Berczeller and Wastl, *Biochem. Z.*, *140*, 368 (1924).
5. Blom, *Farbe u. Lack.* (1927), 329.
6. Blom, *Kolloid-Z.*, *51*, 186 (1930).
7. Boswell, *Trans. Far. Soc.*, *18*, 34 (1922).
8. Boutaric and Semelet, *Rev. Gen. colloides*, *4*, 268 (1926).
9. Briggs, *J. Phys. Chem.*, *34*, 1326 (1930).
10. Coward, *J. Chem. Soc.*, *125*, 1470 (1924).
11. Cunningham, *Proc. Roy. Soc.* (*London*), *83A*, 357 (1910).
12. Deerr, *Intern. Sugar J.*, *22*, 618 (1920).
13. Dhar and Ghosh, *J. Phys. Chem.*, *30*, 628 (1926).

14. Dhar, Sen and Ghosh, *J. Phys. Chem.*, *28*, 457 (1924).
15. Faxen, Dissertation, Upsala, 1921; and *Arkiv. f. Mat., Astron. och Fysik*, *17*, No. 27 (1922).
16. Gans, *Sitz. K. B. Akad. München*, *1911*, 191.
17. Gibbs, *Trans. Inst. Chem. Eng.*, *1933*, 137.
18. Hardy, *Proc. Roy. Soc.*, *66*, 110 (1899); *Z. physik. Chem.*, *33*, 385 (1900); *J. Phys. Chem.*, *4*, 235 (1900).
19. Harkins and Gans, *J. Phys. Chem.*, *36*, 86 (1932).
20. Hartman, "Colloid Chemistry," p. 278, 376 (1939).
21. Il'in, *Z. physik. Chem.*, *122*, 137 (1926); *Chem. Zentr.*, *1926*, II, 3090.
22. Jaeger, *Sitzb. Akad. Wiss. Wien (2a)*, *128*, 1271, 1298 (1919).
23. Kermack, M'Kendrick and Ponder, *Proc. Roy. Soc. Edinburgh*, *49*, 170 (1929).
24. Kermack and Williamson, *Proc. Roy. Soc. Edinburgh*, *47*, 202 (1927).
25. Kruyt and de Jong, *Z. physik. Chem.*, *100*, 250 (1922); *Kolloid. Beihefte*, *28*, 1 (1928).
26. Millikan, *Phys. Rev.*, *32*, 349 (1911).
27. Nordlund, *Z. physik. Chem.*, *87*, 60 (1914).
28. Oden, *Kolloid-Z.*, *26*, 100 (1920).
29. Piechowicz, *Przemysl Chem.*, *18*, 4 (1934).
30. Robinson, *Ind. Eng. Chem.*, *18*, 869 (1926).
31. Rona and György, *Biochem. Z.*, *105*, 133 (1920).
32. Samuel, *Trans. Inst. Chem. Eng.*, *16*, 47 (1938).
33. Schulze, *J. Prakt. Chem.*, *25*, 431 (1882); *27*, 320 (1883); *32*, 390 (1884).
34. von Smoluchowski, *Kolloid-Z.*, *18*, 190 (1916).
35. Steenhoff and Funke, *Tek. Tid. Uppl. C, Kemi*, *65*, 53 (1935).
36. Stewart and Roberts, *Trans. Inst. Chem. Eng.*, *1933*, 124.
37. Stokes, *Transactions*, Cambridge Philosophical Soc., Vol. 9, p. 8 (1851); also *Mathematical and Physical Papers*, Vol. 3, p. 1 (1901).
38. Tiselius, *Kolloid-Z.*, *59*, 306 (1932).
39. Tominaga, *Bull. Inst. Phys. Chem. Research (Tokyo)*, *7*, 298 (1928).
40. Williams, *Trans. Far. Soc.*, *18*, 53 (1922).

Surface per Gram of Cotton Fibres as a Measure of Fibre Fineness

By R. R. SULLIVAN and K. L. HERTEL *

Abstract

It is suggested that fibre surface per gram of fibre be used as a measure of fibre fineness. An air-flow method of quickly and accurately measuring this quantity is described. Either high-porosity ($.984 < \epsilon < .994$) or low-porosity ($.665 < \epsilon < .89$) wads may be used with proper calibration, though wads of intermediate porosity are avoided. The method is checked against determinations by a microscope, and calibrations for two particular pieces of apparatus are given.

Introduction

WHILE "fineness" of fibre is known to be an important property of cotton, an altogether suitable measure of this property has been lacking. The quantity measured should lend itself to quick and accurate evaluation. The quantity should also be one which will suitably distinguish between cottons termed "coarse" and cottons termed "fine." In this paper it is proposed to measure fineness in terms of fibre surface per gram mass, and to show further how this quantity may be quickly and accurately determined. The surface per gram might be converted into an effective diameter; for instance, the diameter of a circular cylinder which would have the same surface per gram. Since the cotton fibre is not circular in cross section, however, this procedure would be artificial. Furthermore, it seems that a measure of the fibre surface would be a suitable measure of fineness for correlating with yarn strength, since the frictional force on a fibre in a yarn is transmitted through the fibre surface. The principle used here for finding surface per gram is similar in some respects to that of Carman [1] for finding specific surface, i.e., surface per unit volume of powders, in that it involves permeability measurements on a porous wad of the material. E. J. Wiggins [2] et al. also have applied the method of Carman to glass, wool, and Celanese fibres. However, since the porosity of a wad of cotton may be changed over a wide range by compression, new features [3] are introduced that do not occur in the case of powders. Since it is not feasible to present here the theory of the flow of air through porous media, it will suffice to point out that under a small pressure gradient the slow flow of air through a low-porosity wad is represented accurately by the relation

$$Q = \frac{A\epsilon^3}{k\mu S_0^2(1-\epsilon)^2}\frac{\Delta P}{L} \qquad (1)$$

where Q = rate of flow in cc/sec, A = area of cross section, ϵ = porosity

* The University of Tennessee Agricultural Experiment Station, Knoxville, Tennessee.

$= \dfrac{\text{interstitial volume of wad}}{\text{total volume of wad}}$, $S_0 = \dfrac{\text{surface of fibre}}{\text{volume of fibre}}$, μ = coefficient of viscosity of air, ΔP = pressure difference across wad, L = length of wad, k = proportionality factor (see later). The relation,

$$S = S_0/\delta \tag{2}$$

where S = surface per gram and δ is the density of the fibre in grams per cc, combined with (*1*) and solved for S, gives

$$S = \sqrt{\frac{A}{\delta^2 k\mu} \frac{\epsilon^3}{(1-\epsilon)^2} \frac{\Delta P}{LQ}} \tag{3}$$

Also, since

$$\epsilon = 1 - \frac{M}{\delta AL},$$

$$S = \sqrt{\frac{\Delta P}{Q} \frac{\left(AL - \dfrac{M}{\delta}\right)^3}{M^2 L^2 k\mu}} \tag{4}$$

where M is the total mass of wad. Now, in (*4*) if all quantities except Q, M and δ are constant the approximate relation,

$$S \doteq \frac{\text{const}}{MQ^{\frac{1}{2}}} \tag{5}$$

is obtained where the degree of approximation depends upon the constancy of k and the constancy of the term $(AL - M/\delta)$. In case of low-porosity wads the constancy of k may be assured by wads being prepared always in the same way. Work by the present authors * shows that the value of k depends upon the orientation of the fibres, being about half as great where fibres are parallel to direction of flow as when they are perpendicular to direction of flow. The quantity k was found to have a value of approximately 6.3 in the part of the present work dealing with low porosity ($.665 < \epsilon < .89$) where long fluffy wads were compressed in the direction of air flow. The process of compression tends to make the fibres perpendicular to the direction of air flow, which in turn would be expected to give a large value for k. Since the values of the density of various cottons are about the same, $(AL - M/\delta)$ will be sensibly constant if approximately the same mass of cotton is used in each case. For wads of higher porosity ($\epsilon > 0.89$) work by the present authors indicates that while relation (*1*) must be altered, yet after a transition period the altered relation holds over a range of high porosities ($.984 < \epsilon < .994$). Thus in case of either high- or low-porosity wads, $(MQ^{\frac{1}{2}})^{-1}$ was calculated for each type of fibre. Since $Q = \dfrac{\text{volume of air}}{\text{time}}$ there was also calculated for a given volume of air $T^{\frac{1}{2}}/M$ for each type of fibre (Table 3). When M is held sufficiently constant, a graph of T against S gives a convenient form of the calibration curve.

For the initial calibration it was necessary to know the densities of the cottons. Now, it is noticed, from equation (*4*), that when two flow measure-

* Reported March 23, 1940, at Charleston, S. C., meeting of the Southeastern Section of the American Physical Society.

ments at constant pressure difference are taken on the same wad of cotton but at different degrees of porosity, there is the relation,

$$\delta = \frac{M}{A}\left\{\frac{(Q_1L_1^2)^{\frac{1}{2}} - (Q_2L_2)^{\frac{1}{2}}}{(Q_1L_1^2)^{\frac{1}{2}}L_2 - (Q_2L_2^2)^{\frac{1}{2}}L_1}\right\} \tag{6}$$

where subscripts 1 and 2 refer to the first and second measurements respectively. It also follows from equation (*4*) that $(QL^2)^{\frac{1}{2}}$ plotted against L gives a straight line. Furthermore, there is the relation,

$$S = \frac{\varphi}{Mm^{\frac{1}{2}}} \tag{7}$$

where

$$\varphi = \left(\frac{\Delta pA^3}{k\mu}\right)^{\frac{1}{2}} \tag{8}$$

and m is the slope of the straight line.

In routine measurements where Δp, A, k and μ are held constant φ also will be constant, so that the surface per gram of fibres will be given in terms of the slope of the straight line and the mass of the wad of fibres. It must be remembered that these relations are valid only over the indicated range of low porosities.

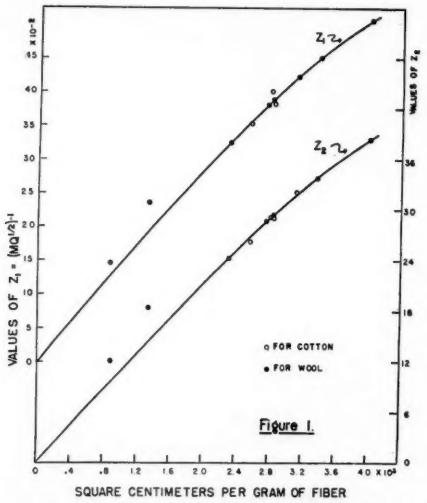


Fig. 1. Curves for Z_1 and Z_2 show these quantities (Table 1) for loosely packed fibers plotted as functions of the fibre surface per gram.

Experimental Procedure

Nine cottons covering a wide range of fineness and staple length were chosen for this investigation. Each was measured by the flow method. Of these nine cottons, five were examined microscopically, five slides of about

TABLE 1

Microscopic Determinations of S_0 for Cottons

Type of fibre	Slide 1		Slide 2		Slide 3		Slide 4		Slide 5		Mean
	No. of Fibres	$S_0 \times 10^{-3}$ cm^{-1}	No. of Fibres	$S_0 \times 10^{-3}$ cm^{-1}	No. of Fibres	$S_0 \times 10^{-3}$ cm^{-1}	No. of Fibres	$S_0 \times 10^{-3}$ cm^{-1}	No. of Fibres	$S_0 \times 10^{-3}$ cm^{-1}	$S_0 \times 10^{-3}$ cm^{-1}
B–38–$\frac{7}{8}$″	541	3.72	520	3.84	497	3.72	564	3.79	621	3.47	3.69
B–38–1$\frac{1}{32}$″	532	3.78	520	3.90	523	3.79	515	3.80	533	3.89	3.83
B–38–1$\frac{3}{16}$″	519	4.38	509	4.48	512	4.58	506	4.08	504	4.53	4.41
Pima	524	4.65	523	4.67	512	4.41	541	4.45	500	4.31	4.50
Sea Island S–38	620	5.32	633	4.88	605	4.61	613	4.98	610	4.88	4.93

Analysis of Variance for Values of S_0 for Cotton

Source of Variation	Sum. of Sq.	D.F.	Mean Square
Between types	5.0998×10^6	4	$1.275^{**} \times 10^6$
Within types	0.6126×10^6	20	0.0306×10^6
Total	5.7124×10^6	24	

Standard deviation per slide $= \sqrt{.0306 \times 10^6} = 0.175 \times 10^3 = 4\%$ of mean value.
Standard error for mean of five slides $= 0.175 \times 10^3 / \sqrt{5} = 0.078 \times 10^3 = 1.8\%$ of mean value.

Additional microscopic values used

Welsh wool — 3194 fibres gave $S_0 = 1.167 \times 10^3$ cm^{-1}
Fine Noil wool — 2871 fibres gave $S_0 = 2.068 \times 10^3$ cm^{-1}

500 fibres each being prepared for each of the five cottons. The fibres were sectioned in a Hardy sectioning device, collodion being used. The sections were then mounted in Canada balsam. By means of a camera lucida attached to the microscope, the image of each fibre was traced on paper. These tracings were then measured for area by a planimeter and for perimeter by a map measure. S_0 was then given by

$$S_0 = \frac{\text{Sum of perimeters}}{\text{Sum of areas}}$$

Results of this work are given in Table 1. An analysis of variance for the results of the 25 slides is also given. This indicates a standard error of the mean of five slides to be 1.8% of the mean S_0.

For the flow data, copper tubes fitted with pistons were used. The cotton, after being rendered fluffy by a carding device and conditioned by air being drawn through it, was placed in the tube and compressed to the desired porosity by the piston. Air was drawn through the wad by mineral oil flowing from a gallon bottle to which the cylinder was attached. Pressure drop was measured with a kerosene manometer and a traveling microscope. Pressure drop was held constant by regulation of the flow of oil. Q, the rate of flow, was obtained by noting, with a stop watch, the time necessary for the oil to fill a volumetric flask. The data for density determinations and slope determinations for equations (*6*) and (*7*) were taken under standard conditions of temperature and humidity (65% R.H. 70° F), while three sets of determinations, for use in equation (*5*), were made with ordinary laboratory air. Relative agreement between these different methods was good.

Results

Table 2 gives results from the two-point determinations on 1.5-gm samples used with equations (*6*) and (*7*). Values of S_0 from Table 1 divided by values of density from equation (*6*) give the values of surface per gram listed in the fifth column. These values, when multiplied by corresponding values of $M\,m^{\frac{1}{2}}$ from the two-point determinations, give values for the φ of equation (*7*). The seventh column of the table shows values of S for all types of fibre studied, the values being obtained by dividing the weighted mean φ of equation (*7*) by the respective two-point determinations of $M\,m^{\frac{1}{2}}$. These values of S are subsequently taken as the values of surface per gram for the given types and are used for calibrating the single-point determinations. The accompanying analyses of variance indicate the standard errors of the quantities involved in Table 2. As is seen, the uncertainty of the relative values of S is represented by a standard error of about 1% of the mean (obtained from analysis of $M\,m^{\frac{1}{2}}$ data), while the uncertainty in the absolute values of S is represented by a standard error of about 3% of the absolute values (obtained from standard error of φ and of $M\,m^{\frac{1}{2}}$). Table 3 gives values for single-point determinations for both high and low porosities. Z_1 and Z_2 are for high porosities, Z_3 and Z_4 are for low porosities. Figs. 1 and 2 show values of Z_1, Z_2, Z_3, Z_4 of Table 3 plotted against corresponding values of S from Table 2. It is seen from the linearity of the curve for Z_3, and from the smoothness of curves for Z_1 and Z_2, that the variation of density among the cottons is small enough to be neglected in these calibration curves. It is seen also that the densities of the wools used are sufficiently different from those of the cottons to cause the points for wool to be off the curves for cotton. In case

TABLE 2

Results for Two-Point Flow Determinations

Type of Fiber	No. of determinations	Mean δ gm/cc	$M\,m^{\frac{1}{3}}$	S_0/δ $cm^2/gm \times 10^{-3}$	$\varphi \times 10^3$	$S = \frac{3.46}{M\,m^{\frac{1}{3}}}$ $cm^2/gm \times 10^{-3}$
B–38–$\frac{7}{8}$″	30	1.59	1.499	2.32	3.48	2.31
B–38–1$\frac{1}{32}$″	30	1.57	1.348	2.44	3.29	2.57
B–38–1$\frac{3}{16}$″	30	1.53	1.254	2.88	3.74	2.76
Pima	30	1.54	1.110	2.92	3.24	3.12
Sea Island S–38	30	1.53	1.025	3.22	3.30	3.38
Mean φ for the five cottons = 3.41						
Standard error of mean = .093						
B–38–S–1$\frac{3}{16}$″	10	1.54	1.217			2.84
Sea Island–C–38	10	1.54	0.865			4.00
Sacaton No. 7	10	1.56	1.239			2.80
Hopi	7	1.52	1.221			2.83
Welsh Wool	10	1.283	3.887	0.910	3.54	0.890
Fine Wool Noil	10	1.315	2.580	1.574	4.07	1.341

Weighted mean constant for cotton and wool = 3.46

Analysis of variance for δ (From first five types of fibre)

Source of variation	Sum of sq.	D.F.	Mean square	F	$F_{req.}$ (1% level)
Between types	0.135662	4	.033916*	5.31	3.44
Within types	0.924507	145	.006376		
Total	1.060169	149			

Standard deviation per individual = $\sqrt{.006376}$ = .0798

Standard error for mean of 10 = $.0798/\sqrt{10}$ = .0252 = 1.6%

Standard error for mean of 30 = $.0798/\sqrt{30}$ = .0145 = 1%

Analysis of variance for $M\,m^{\frac{1}{3}}$ (From first five types of fibre)

Source of variation	Sum of sq.	D.F.	Mean square
Between types	4.252742	4	1.0632
Within types	0.246242	145	0.001698
Total	4.498984	149	

Standard deviation per individual = $\sqrt{.001698}$ = 0.0412

Standard error for mean of 10 = $0.0412/\sqrt{10}$ = 0.013 = 1.0%

Standard error for mean of 30 = $0.0412/\sqrt{30}$ = 0.00752 = 0.6%

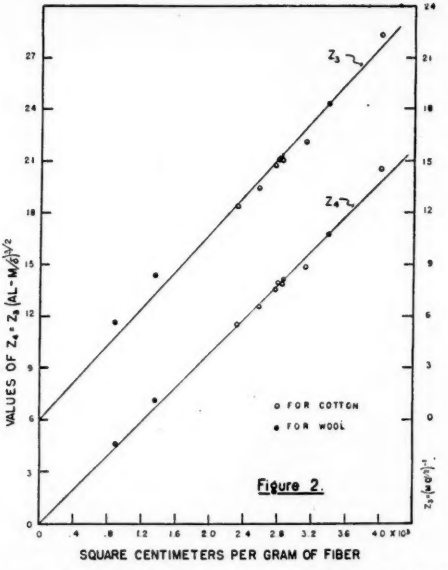


Fig. 2. Curves for Z_3 and Z_4 show these quantities (Table 3) for tightly packed fibres plotted as functions of the fibre surface per gram.

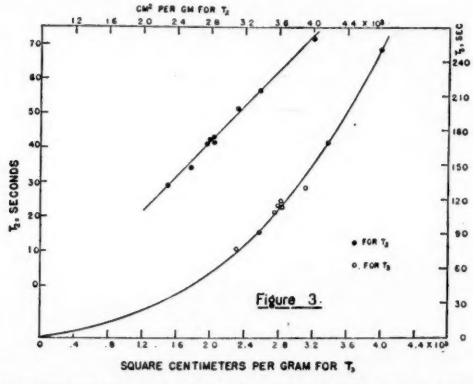


Fig. 3. Calibration Curves for Obtaining Cotton Fibre Fineness. The curve for T_2 shows relationship between fibre surface per gram and the time in seconds required for a head of 79.8 dynes/cm² to cause 500 cc of air to flow through a 0.1-gm wad of fibres packed to a length of 1.76 cm in a tube of 1.993 cm inside diameter. The curve for T_3 shows relationship between fibre surface per gram and the time in seconds required for a head of 1196 dynes/cm² to cause 50 cc of air to flow through a 0.1-gm wad of fibres packed to a length of 0.5118 cm in a tube of 0.8165 cm inside diameter.

of Z_4, where correction for density has been applied, the wools and cottons, within limits of error, lie on the same curve. If corrected for density, a single calibration curve would probably suffice for fibres of different densities or if desirable separate calibration curves could be used for fibres of various densi-

TABLE 3

Results for Single-Point Flow Determinations

Type of Fiber	$Z_1 \times 10^2$	Z_2	Z_3	Z_4	T_2 sec for M = 0.1	T_3 sec for M = 0.1
B–38–$\frac{7}{8}$"	3.24	2.413	12.38	1.152	29.1	76.6
B–38–1$\frac{1}{32}$"	3.52	2.614	13.48	1.255	34.2	91.1
B–38–1$\frac{3}{16}$"	3.80	2.867	14.78	1.358	41.1	109.2
Pima	4.23	3.202	16.15	1.488	51.3	130.4
Sea Island S–38	4.51	3.363	18.37	1.677	56.5	168.7
B–38–S–1$\frac{3}{16}$"	3.82	2.882	15.08	1.389	41.5	113.7
Sea Island C–38	5.07	3.835	22.32	2.053	73.5	249.1
Sacaton No. 7	4.00	2.909	15.11	1.391	42.3	114.2
Hopi	3.82	2.934	15.35	1.400	43.0	117.8
Welsh Wool	1.45	1.205	5.67	0.462		
Fine Noil Wool	2.35	1.835	8.45	0.718		

$Z_1 = (MQ^{\frac{1}{2}})^{-1}$ for 8.0-gm plugs in tube of 3.88 cm diameter and 38.06 cm long with $\Delta p = 398.67$ dynes/cm².
$Z_2 = (MQ^{\frac{1}{2}})^{-1}$ for 0.1-gm plugs in tube of 1.993 cm diameter and 1.76 cm long with $\Delta p = 79.8$ dynes/cm².
$Z_3 = (MQ^{\frac{1}{2}})^{-1}$ for 0.1-gm plugs in tube of 0.8165 cm diameter and 0.5118 cm long with $\Delta p = 1196.0$ dynes/cm².
$Z_4 = Z_3(AL - M/\delta)^{\frac{1}{2}}$ where values of δ were obtained from Table 2.
T_2 = mean time in seconds for flow of 500 cc in case of Z_2.
T_3 = mean time in seconds for flow of 50 cc in case of Z_3.

Both for determining Z_2 and Z_3, three samples of each type of fibre were prepared, two determinations being made on each sample. Thus the sensitivity of the methods may be determined.

Analysis of Variance for Z_2, Z_3, Z_4 for Cottons

Source of variation	D. F.	Mean square		
		Z_2	Z_3	Z_4
Between types	8	1.054	51.299	0.4187
Samples within types	18	.00378	0.1055	0.000687
Det. within samples (Error)	27	.0000287	0.00494	0.0000505
Standard deviation of determination		.00536 = 0.18%	.0702 = 0.44%	0.00711 = 0.48%
Standard deviation of sample		.065 = 2.2 %	.325 = 2.04%	0.0262 = 1.8 %
Standard error of mean of 3 samples		.0375 = 1.2 %	.1875 = 1.2 %	0.0151 = 1.03%

ties. In the present paper, however, the wools were added chiefly to show the nature of the correction for density. From the analysis of variance of Table 3 for cottons and from Fig. 2 it is noted, by comparison of Z_3 and Z_4, that no great increase in accuracy for cottons is given by the correction for density. In either case the standard error of the determination (for Z_2, Z_3, or Z_4) expressed in percent is less than the standard error for the calibration (S of Table 2) expressed in percent.

Fig. 3 gives two calibration curves for single-point determinations of S, one curve being for high and one for low porosity. These curves were prepared by plotting T_2 and T_3 of Table 3 against S of Table 2. The curves then were checked by actual use. Thus, to find S for a cotton, 0.1 gram of fibre is fluffed on a carding device, placed in the copper tube of 0.8165 cm inside diameter and compressed to 0.5118 cm long. The time required for a head of 1196 dynes/cm² to cause 50 cc of air to flow through the wad is taken by a stop watch.* Then, corresponding to this time, the value of S is read from curve for T_3. Similarly 0.1 gram of cotton may be fluffed on a carding device, placed in the 1.993-cm diameter tube and compressed to 1.76 cm. The time then taken for a head of 79.8 dynes/cm² to cause 500 cc of air to flow may be used with curve for T_2 to give S.

The use of the curve for T_3 has the advantage of involving a more easily measured head than that required for T_2. The use of curve for T_2 has the advantage that fluctuations of external atmospheric pressure are less troublesome. The accuracy of the two methods, however, is found to be about the same as is noted from the analyses of Table 3.

Summary

It was suggested that surface per gram is a suitable measure of fineness for cotton fibres. An air-flow method of determining this quantity to within a standard error of 3% was described and two calibration curves, one for high- and one for low-porosity wads of fibre, were given. Analyses were given which indicated that the mean from single air-flow tests on each of three 0.1-gm wads gave the same accuracy as did the mean from microscopic measurements of 2500 individual fibres.

The authors wish to acknowledge here the assistance of Miss Elizabeth Cotter, Miss Louise McCamy, and Miss Beth Cheney, who took much of the set of data used for the calibrations leading to values of S in Table 2.

References

1. Carman, P. C., *Trans. Inst. Chem. Eng.*, *15*, pp. 150–166 (1937); *16*, pp. 168–188; also *J. Soc. Chem. Ind.*, *57*, pp. 225–235 (1938); *58*, pp. 1–7 (1939).
2. Wiggins, Campbell, and Maass, *J. Canadian Res.*, *17*, p. 318 (1939).
3. Fowler and Hertel, *J. Applied Physics*, *11*, pp. 496–502 (1940).

* Pressure drops given here are for cotton wad only and do not include any additional drop across the restraining ends of the tube.

X-ray Studies of Silk Fibres

By H. J. McNICHOLAS *

Abstract

X-ray studies were made of a Bombyx mori type of silk and of a Tussah silk. Differences in the diffractive properties of the fibroin components of these silks are recorded. Diffraction bands were found for the sericin components of each type of silk, showing that this component also exists on the fibre in a partially crystalline condition but with a lower degree of crystallite orientation than is exhibited by the fibroin component. The positions of the observed sericin bands are different for each type of silk. Comparison of the diffraction patterns of hard and soft silks of the Bombyx mori type showed no significant difference between them.

Introduction

THE diffraction of x-rays by natural silk fibres reveals the presence of a high degree of regularity in the arrangement of atomic or molecular units in parts of the fibre structure. The observed pattern of interference maxima as usually recorded on a photographic film may be explained on the assumption that the fibre contains many minute crystallites, or nuclei of crystallization, which may vary in structure, composition, number, size, and orientation. This approach to a crystalline organization of the fibrous material, and the changes accompanying various chemical treatments, has been studied by several investigators[1, 2, 3, 4, 5, 6, 7] and will be considered below in relation to the present work.

Materials and Method

The x-ray studies were made of a *Bombyx mori* type of silk and of a *Tussah* silk. The silks were dewaxed by two cold extractions, for 24 hours each, with alcohol and ether, respectively. The sericin and fibroin components of the *Bombyx mori* silk were separated by heating the dewaxed silk for 15 minutes in an autoclave at 115° C. After evaporating the water solution to a thick syrup the sericin was precipitated by adding it slowly to ice-cold acetone. For the purpose of comparing the diffractive properties of the two types of silk fibroin the dewaxed samples were degummed by three successive treatments for ½ hour each with boiling 1% solution of neutral soap, followed by a thorough washing with water.

The author is indebted to H. A. Rutherford, of the Textile Foundation Research Associateship at the National Bureau of Standards, for the preparation of the fibres used in this work.

The source of x-rays was a hot-filament copper-target three-window tube operated at 15–20 ma and 35 peak kv. Each beam of x-rays was

* Physicist at the National Bureau of Standards.

defined by a cylindrical hole 0.7 mm in diameter drilled axially in a brass rod 40 mm long. Additional apertures for the reduction of scattering were employed, and filters of nickel foil 0.025 mm in thickness were mounted over each window. The patterns were recorded on a flat film perpendicular to the x-ray beam at a distance of 51.5 mm from the sample.

Compact fibre bundles 1 to 1.5 mm in diameter were prepared with the aid of a Chandler bundle wrapping machine.[8] The fibres were combed approximately parallel, the ends were tied and clamped in the jaws of the machine, and the bundle was held under a tension of 5 pounds while being wrapped with one layer of cotton thread. The fibres were moistened with a drop of distilled water before wrapping and then allowed to dry under tension. Before mounting in the diffraction camera the layer of cotton thread was removed leaving a compact stick-like bundle of fibres which could be centered accurately in the x-ray beam with the axis of the bundle perpendicular to the beam.

For the determination of the perpendicular distance from the fibre bundle to the film, bundles were made up in the above manner from cotton fibres thoroughly soaked in concentrated aqueous NaCl solution. When dry the salt was deposited in finely divided crystalline form throughout the fibre bundle and hence gave the ring pattern of NaCl superposed on the weaker spot pattern of the fibres. From the known diffraction angles for the NaCl bands, and the observed diameters of the corresponding rings, the required distance was determined.

The positions of the interference maxima * on the film were determined by placing the film on an illuminated ground glass plate and spotting the maxima with pen and ink, then measuring distances between spots with a millimeter scale. From these measurements and the known distance from sample to film the values of tan 2θ and cos δ_0 were computed for each interference maximum, where 2θ is the angle between the diffracted beam and the central undeviated beam and δ_0 is the angular displacement of the maximum from the meridian line on the film. The interplanar spacings,[9] d, were derived from the Bragg relation. The identity period,[9] b, along the fibre axis, and the cylindrical coordinates (ζ, ξ) of position in the Ewald[10] reciprocal lattice, were also computed and tabulated by the methods described by Bernal.[11]

Sericin and Fibroin Components of Raw Silk

The interplanar spacings, d, corresponding to the most prominent and sharply defined interference maxima on the diffraction patterns of a *Bombyx mori* type of silk, in the raw, dewaxed, and degummed conditions, are given in Table 1. These bands are not changed appreciably in position or relative intensity after the removal of the wax and sericin components. The patterns for the raw and dewaxed silks, however, show the presence of some weak interference bands, not included in the table, with d-values of approximately 5.2, 4.6, 4.1, and 3.8 angstroms, which are not present in the

* The designation of the maxima follows an established custom. The positions of the maxima (spots) on the fibre diagram (see Fig. 1) are symmetrical with respect to two mutually perpendicular axes through the center, called the meridian and equator lines, respectively. The meridian (vertical axis in Fig. 1) is parallel to the long axis of the fibre bundle. The centers of the spots, or arcs, lie on concentric hyperbolae, called layer lines, having the meridian line for their common axis. The notation A, I, II, III, etc., refers to the equator, 1st layer line, etc., and numerical subscripts refer to successive spots on a layer line, progressing outward from the meridian.

TABLE 1

Comparison of the diffraction patterns of raw, dewaxed, and degummed silks, and of raw silks of hard, medium, and soft nature.

Numbers given are interplanar spacings, *d*, in angstroms, corresponding to the most sharply defined interference maxima on the fibre diagram. Each value is a mean *d* for three or more different samples.

Interference maximum	A_2	A_3	A_4	I_2	I_3	II_1	II_2	III_1
Raw silk	4.77	4.27	3.08	3.63	2.22	3.27	2.77	2.05
Dewaxed	4.79	4.27	3.06	3.64	2.22	3.26	2.77	2.06
Degummed	4.73	4.27	3.05	3.63	2.23	3.28	2.76	2.05
Raw Silk								
Hard	4.76	4.27	3.06	3.64	2.22	3.28	2.77	2.05
Medium	4.78	4.28	3.08	3.64	2.23	3.28	2.76	2.05
Soft	4.77	4.26	3.06	3.63	2.22	3.29	2.77	2.06

degummed silks and appear with equal intensity for the raw and dewaxed conditions. These bands, therefore, may be assigned to the sericin on the raw silk, and the remaining bands to the fibroin component. Any bands arising from the wax itself are too weak to show or are completely masked by the other materials.

Some weak bands of similar appearance are also observed on the patterns of raw *Tussah* silk, and may be assigned for the same reasons to the sericin component. They differ in number and position, however, having d-values of approximately 5.9, 3.6, 3.0, and 2.4 angstroms.

The presence of sericin bands on the patterns for both silks indicates some degree of crystallinity in the sericin as it exists on the native fibre, but the bands are spread over circular arcs of larger angular extent than the arcs (maxima) for the fibroin component. Since the degree of unidirectional orientation of the crystallites is indicated by the extent to which the circular diffraction bands are concentrated into arcs, these observations show that the preferred orientation of crystallites with respect to the fibre axis is less pronounced in the sericin than it is in the fibroin component.

The sericin was separated from the *Bombyx mori* fibres by the method previously described. The interplanar spacings for the regenerated sericin are not all the same as for the original material as it exists on the fibre. Bands now appear with d-values of 7.3, 4.6, 3.8, 2.3, and 2.1 angstroms. This result may be a consequence of the hydrolysis of sericin, which has been shown by Rutherford and Harris [12] to occur during the degumming process. In contrast with this behavior of sericin, it has been shown [5, 6] that the spacings in regenerated silk fibroin are the same as in the original material.

Raw Silks of Hard and Soft Nature

Raw silks of the same type may exhibit small differences in physical characteristics [13] which are frequently described by the terms "hardness" and "softness." Little is known about the intrinsic physical or chemical differences between these silks, and it was desired to know whether any significant difference would appear in their diffraction patterns.

Three fibre bundles were made of each of the hard, medium, and soft classifications of *Bombyx mori* silk and the diffraction patterns compared. In appearance there was no significant difference between them. The sericin bands appeared the same, and measurements of the position of the most sharply defined interference maxima of the fibroin component gave interplanar spacings as recorded in Table 1. These measurements, however, refer only to the structure of the crystallite and do not determine any difference that may exist in the orientation of crystallites or in the degree of crystallization. It can be said only that any differences of this nature must be too small to make any appreciable difference in the general appearance of the diffraction patterns. More refined methods for measuring the variations in density distribution over the pattern may lead to the evaluation of significant differences between the hard and soft silks.

Comparison of Silks from Different Sources

Comparison [1, 3, 4, 6] of the diffraction patterns of degummed silks from different sources has shown differences in the positions and intensities of interference maxima on the fibre diagram. Trogus and Hess [4] pointed out that the positions were the same for some of the maxima but differed definitely for others. They suggested the possibility of two or more different crystalline components in the fibroin, one of which was common to all the silks. Kratky and Kuriyama,[3] on the other hand, suggested that only one kind of crystalline material is present in the fibroin component of a given silk, and placed all silks examined into two distinct groups, each group having a characteristic diffraction pattern.

All data at present available are perhaps not sufficiently extensive and accurate to determine how closely all silks may be classified into groups by their diffraction patterns. In the present paper this deficiency is not removed, but further data are given on a *Bombyx mori* type of silk and on a *Tussah silk*, which exhibit marked differences in diffractive properties. The patterns are shown in Fig. 1, and measurements of the positions of the interference maxima and derived data are recorded in Table 2.

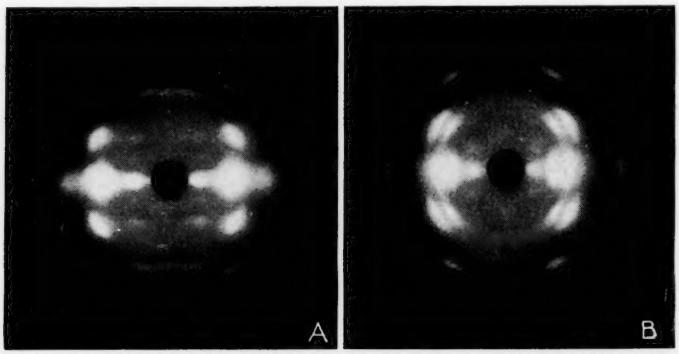


Fig. 1. X-ray diffraction patterns of degummed silk fibres. A, *Bombyx mori*. B, *Tussah*.

TABLE 2

Comparison of x-ray diffraction data for *Bombyx mori* and *Tussah* silks.

2θ = angle of diffraction measured from central undeviated beam.
δ_0 = angular distance of interference maximum from meridian line of pattern.
(ζ, ξ) = cylindrical coordinates in reciprocal lattice.
d = interplanar spacing.
b = identity period along fibre axis.
Letters m, v, s, w in the intensity column are abbreviations for words, medium, very, strong, weak, respectively.

Layer line	Interference maximum	Intensity	tan 2θ	cos δ_0	ζ	ξ	d angstroms	b angstroms
Bombyx mori (Mean values from 22 diffraction patterns)								
A	A_1	ms	0.177	0.000	0.000	0.175	8.8	—
	A_2	w	.335	.000	.000	.322	4.78	—
	A_3	vs	.381	.000	.000	.361	4.27	—
	A_4	w	.559	.000	.000	.504	3.06	—
	A_5	vw	.786	.000	.000	.654	2.36	—
I	I_1	w	.282	.804	.218	.166	5.61	7.06
	I_2	s	.456	.535	.222	.362	3.63	6.94
	I_3	vw	.852	.339	.220	.655	2.23	7.01
II	II_0	w	.474	1.000	—	—	3.48	7.18
	II_1	ms	.513	0.965	.440	.163	3.28	6.99
	II_2	w	.632	.828	.442	.337	2.77	6.96
	II_3	vvw	.823	.694	.441	.511	2.28	6.99
III	III_0	vw	.837	1.000	—	—	2.26	7.19
	III_1	w	.966	0.940	.654	.367	2.05	7.07
							Mean	7.04
Tussah (Mean values from 15 diffraction patterns)								
A	A_1	ms	0.179	0.000	0.000	0.178	8.7	—
	A_2	vs	.298	.000	.000	.289	5.33	—
	A_3	vs	.371	.000	.000	.353	4.36	—
	A_4	w	.671	.000	.000	.582	2.64	—
	A_5	vw	.780	.000	.000	.649	2.37	—
I	I_1	ms	.382	.609	.218	.290	4.24	7.08
	I_2	s	.448	.543	.222	.354	3.68	6.92
	I_3	vw	.842	.350	.224	.648	2.24	6.84
II	II_0	w	.491	1.000	—	—	3.37	6.93
	II_2	ms	.589	0.871	.444	.285	2.92	6.96
	II_3	vvw	.814	.678	.429	.516	2.30	7.19
III	III_0	vw	.838	1.000	—	—	2.25	7.19
	III_1	w	.962	0.936	.650	.370	2.06	7.12
							Mean	7.03

The data in Table 2 show that the ξ-coordinate in the reciprocal lattice, which distinguishes the layer lines on the observed diffraction pattern, is approximately the same for the corresponding layer line of each type of silk. Hence, the identity period, b, along the fibre axis is the same for each type of silk and equal approximately to 7.0 Angstroms.

It should be noted in this connection that there is one weak interference maximum, not listed in Table 2, which is located between the equator and first layer line, and near the center of the pattern for each type of silk. Its presence suggests the possibility that the true layer line system for these silks may consist of alternate layers of strong and very weak spots, only the stronger alternate layers being readily observed in this work. If this is the true situation then the fibre period, b, would be double that given above. Measurements of the position of the interference maximum in question, on the assumption that it lies on the first layer line of the suggested system, give a b value of approximately 12.8 for each type of silk. This is lower than the required value but more accurate measurements should be made to settle this point.

The ξ-coordinates for the observed interference maxima are the same within experimental error for spots III_1, II_2, I_2, I_2, A_2, and A_1, but differ for spots II_2, II_1, I_1, A_4, A_3, and A_2 by amounts beyond the uncertainty of the measurements. It would be of interest to extend such measurements to silks from many different sources to see if all the differences lie only in the ξ-coordinate, as would be expected if the silks differed in the nature of side-chains attachments to main polypeptide linkages along the fibre axis.

References

1. R. Brill. *Liebig's Ann. der Chem., 434,* 204 (1923).
2. O. Kratky. *Zeit. Phys. Chem., B5,* 297 (1929).
3. O. Kratky and S. Kuriyama. *Zeit. Phys. Chem., B11,* 363 (1931).
4. C. Trogus and K. Hess. *Biochem. Zeit., 260,* 376 (1933).
5. K. Meyer and H. Mark. *Ber., 61,* 1932 (1928).
6. I. Sakurada and K. Hutino. *Sci. Pap. Inst. of Phys. and Chem. Research,* Tokyo, *21,* 266 (1933).
7. I. Sakurada and Y. Matsushita. *J. Soc. Chem. Ind.,* Japan, Supp. Binding, *40,* 58B (1937).
8. H. B. Richardson, T. L. Bailey, Jr. and C. M. Conrad. *U. S. Dept. Agric. Tech. Bull.* No. 545 (1937)
9. For information on the transformation of measurements of the position of interference maxima on the fibre diagram into corresponding geometrical relations in a crystal lattice, see W. P. Davey, "A study of crystal structure and its applications," 1st Edition (1934), Chapter VII; W. T. Astbury, "Fundamentals of fibre structure" (1933); W. A. Sisson, "X-ray diffraction analysis and its application to the study of plant constituents," *Contributions from Boyce Thompson Institute, 9,* 381 (1938).
10. P. P. Ewald. *Zeit. Kryst., 56,* 129 (1921).
11. J. D. Bernal. *Proc. Roy. Soc.* (London), *A113,* 117 (1926).
12. H. A. Rutherford and M. Harris. *Text. Rsch., 10,* 221 (1940); *J. Research NBS, 24,* 415 (1940).
13. W. P. Seem. *Text. Wld., 80,* 1774 (1930).

Abstracts

Committee on Abstracts and Bibliography:
E. R. Schwarz, Chm., Dr. E. J. Saxl, C. H. Clark.

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I. Fibres (Synthetic and Natural) and Fibre Analysis

Cellulose Fibres; Sorption of Water by. R. S. Neiman and V. A. Kargin. *Prom. Org. Chim.*, 1939, V. 6, P. 628–633 (through *Brit. Chem. Physiol. Abstr.*, 1940, B. 268); abs. in *J. T. I.*, June 1940, V. 31, P. A332.

Sorption and desorption of water vapor are accelerated at low pressures, equilibrium being attained within 1–1.5 hours (McBain's method) as compared with 2–3 months (Obermiller's method). Hysteresis is a feature of both methods. Fibres of equal serimetric index give identical sorption isotherms. The tensile strength per unit area of cross-section of cotton fibre falls with rising moisture content. The hygroscopicity of cotton and sulphite cellulose rises as a result of mercerization, but is unchanged by other processes affecting the length of cellulose units or their orientation. Hygroscopicity is least for cellulose acetate, and greatest for casein fibres. (C)

Crystal Orientation in Cellulose Fibres. E. Plötze and H. Person. *Z. physik. Chem.*, 1940, V. B45, P. 193–200; cf. *C. A.*, V. 24, Col. 3002; abs. in *C. A.*, 1940, V. 34, Col. 4900.

Microphotometer curves were obtained from Debye x-ray photographs of cellulose fibres. The half-angle is independent of the av. degree of polymerization and is 15° for ramie fibres and 32° for cotton. This fact will be useful in a detn. of the factors that det. the strength of fibres. (C)

THE DENSITY AND SWELLING OF SILK FILAMENTS IN RELATION TO MOISTURE CONTENT. A. C. Goodings and L. H. Turl. (Ontario Research Foundation, Canada). *J. T. I.*, June 1940, V. 31, P. T69–T79.

The regain of degummed, white Japanese silk was measured at 25° C. under both adsorption and desorption conditions for relative humidities ranging from dryness to saturation. A value of 35.8% regain for saturated silk was deduced from density and swelling data. Values for the density of the same degummed Japanese silk at moisture contents ranging from dryness to saturation were determined. The longitudinal swelling of silk filaments accompanying moisture absorption were measured. The lateral swelling of silk was computed from the measured longitudinal swelling and the volume swelling derived from density data. (C)

FINE STRUCTURE OF PHLOEM FIBRES. I. UNTREATED AND SWOLLEN HEMP. B. C. Kundu and R. D. Preston. *Proc. Roy. Soc.* (London), 1940, V. B128, P. 214–31; abs. in *C. A.*, 1940, V. 34, Col. 4915.

"In hemp we have the clearest evidence that the bulk of the primary wall is composed of longitudinally directed cellulose chains. It cannot be denied, however, that in this tenuous wall there may occur layers (perhaps in a mol. sense) whose chain direction is different." 33 references. (C)

INVESTIGATION OF THE RELATION BETWEEN THE COMPOSITION OF FLAX AND THE EASE WITH WHICH IT CAN BE BLEACHED. E. A. Ivannikova and S. K. Yakubenko. *L'no-Pen'ko-Dzhutovaya Prom.*, 1938, V. 8, No. 2, P. 32–7; *Chem. Zentr.*, 1938, II, P. 1701; abs. in *C. A.*, 1940, V. 34, Col. 4915.

Fibres with a high lignin content are compact, swell but little and are therefore more difficult to bleach and dye than fibres which contain but little lignin. No direct relation could be established between the chem. compn. of the raw and bleached spun rayon. In one type of flax (in the Rjev region), which is readily bleached, the raw fibre has a high Mn content (0.0054%) and it is assumed that the good bleaching action is due to the catalytic action of the Mn. (C)

THE MICELLAR STRUCTURE OF NATIVE CELLULOSE. O. Kratky, K. Kainz and R. Treer. *Holz. roh u. Werkstoff*, V. 2, P. 409–13 (1939); abs. in *C. A.*, 1940, V. 34, Col. 4899.

Indications regarding the cavities in the micellar system are obtained by swelling the fibre with solns. of salts of noble metals and then depositing the metal by reduction. Canal-like intermicellar cavities seem to run in the direction of the fibre. In ramie the cryst. portion may comprise one-third of the total. The metal-laden fibres show an intensified diffuse blackening which is useful in studying micellar structure. The relation of intermicellar spaces and mech. properties is discussed. (C)

NATIVE AND REGENERATED CELLULOSE. *Silk & Rayon Wld.*, June 1940, V. 17, P. 23–6.

This final article of a series of three deals with the relationship between native and regenerated cellulose and summarizes the modern view of the structure of cellulose. (C)

II. Yarns and Fabrics

The Carpet Trade Needs Rayon—Is Rayon Ready? Jos. Alexander Truitt. *Rayon Text. Mo.*, January 1940, V. 21, P. 73, 74, 77; February 1940, V. 21, P. 63–5. (C)

Comparative Investigations of the Influence of Humidity on the Stretch and Resistance to Tearing of Fabrics. Ernst Thiemann. *Mell. Textilber.*, V. 21, P. 61–4 (1940); abs. in *C. A.*, V. 34, Col. 3504 (1940).

Yarns made from wool (I), hair (II), silk (III), cotton (IV), linen (V), hemp (VI), jute (VII), ramie (VIII), kapok (IX), paper (X), "Vistra" spun rayon (XI), Cu-ammonia rayon (XII), viscose rayon (XIII) and acetate rayon (XIV) were brought to equil. with atms. of varying humidities and subjected to tensile strength and ultimate elongation tests. With increasing humidity the tensile strength of I, II, III, XI, XII, XIII and XIV progressively decreases and the ultimate elongation progressively increases. Both the tensile strength and the ultimate elongation of IV, V, VI, VIII and IX increase. With VII the strength and elongation both increase to a max. at about 70–80% relative humidity, and then decrease sharply. With X the strength and elongation remain roughly const. up to 60–70% relative humidity. From this point on the former decreases and the latter increases sharply. Results are presented in the form of diagrams. (C)

Die Cone Guide for Twine Twisters. By Fairbairn, Lawson, Combe Barbour Ltd. *Text. Mfr.*, July 1940, V. 66, P. 282.

Higher speeds of production and better build of twines or cords are obtained by controlling twist between delivery rollers and flyer, with guide patented. (C)

An Entirely New Principle for the Washing of Fabrics. *Silk J. & Rayon Wld.*, Oct. 1939, V. 13, P. 815–6, 818.

Describes a washing machine in which the cloth passes at its full width between two pressure chambers which extend across the full width of the machine. The pressure chambers are in the form of venturi-shaped slots or nozzles through which the liquid is forced at high velocity in the form of two equally opposed streams which impinge on the passing cloth at the same point. As these jets are equal and opposite, they arrest each other, thereby changing their velocity into pressure, without putting any tension on the cloth. Consequently the fibres of the cloth at this point are virtually washed under pressure. (C)

Factors Influencing Wool Felting. Joseph W. Creely and George Le Compte. *Am. Dye. Rptr.*, 1940, V. 29, P. 292–94, 306.

The authors give a brief outline of factors influencing wool felting and references to the literature of these subjects. They then decided that the following subjects could be profitably studied: The use of felting aids; study of chlorine and its derivatives to see if it is possible both to increase and decrease the feltability of wool; study of the effect of alkalies on felting; study of the effect of a lyotropic series of ions in the felting medium on swelling and on felting; study of wetting agents with respect to wetting

and felting characteristics; study of the effect of carbonizing on the feltability of wool; study of the effect of reducing agents such as bisulfites, hydrosulfites, formaldehyde, etc., on the feltability of wool. They stated that work is now in progress along some of these lines. (C)

FINISHING SPUN RAYON FABRICS. W. A. Wardell. Cotton, July 1940, V. 104, P. 110–13. (C)

INFLUENCE OF SIZES ON FABRIC SERVICEABILITY. H. H. Mosher. *Text. Age*, June 1940, V. 4, P. 48–53. (C)

INFLUENCE OF TWIST ON SHORTENING, DIAMETER, DULLNESS AND TORSION OF SILK HOSIERY YARNS. Warren P. Seem, T. Andrew and E. Simmat. *Rayon Text. Mo.*, February 1940, V. 21, P. 66–8; March 1940, V. 21, P. 72–4; April 1940, V. 21, P. 65–7; May 1940, V. 21, P. 71–3; June 1940, V. 21, P. 67–8; July 1940, V. 21, P. 63–4; Aug. 1940, V. 21, P. 43–4.

These articles deal with the definitions and nomenclature of silk hosiery yarns. Formulas and deductions form valuable data, which can be applied to rayon and the new synthetic yarns, if modified by actual tests. The information obtained by careful tests is of value in planning new fabrics, making comparisons and estimating yarn costs. (C)

LATEST DEVELOPMENTS IN TEXTILE PROCESSING AND FINISHING. N. L. Deutsch. *Rayon Text. Mo.*, April 1940, V. 21, P. 81–2; May 1940, V. 21, P. 78–9.

Discusses new chemical products and new methods developed especially for synthetic fibres, but in many cases adapted for natural fibres. (C)

LOW-TEMPERATURE MERCERIZATION OF COTTON CELLULOSE. T. I. Rudneva and N. I. Nikitin. *Org. Chem. Ind.* (U. S. S. R.) V. 6, P. 398–400 (1939); abs. in *C. A.*, V. 34, Col. 3505 (1940).

Swelling of cotton fibres, with consequent increase in their reactivity, is enhanced by freezing the suspension of cotton in aq. NaOH. The greatest effect is obtained in 8% NaOH at —15°. (C)

MOLDS: CONDITIONS OF DEVELOPMENT, IDENTIFICATION OF FAULTS OF CELLULOSE FABRICS AND REMEDIES. Joseph Riere. *Rev. gén. mat. color.*, V. 43, P. 441–4 (1939); abs. in *C. A.*, V. 34, Col. 3505 (1940).

The action of molds on cellulose is considered to be depolymerization. Cellulose, which is highly polymerized, is not attacked by species of *Mucor* and *Aspergillus*, but hydrocellulose, etc., are attacked. Various antiseptic compds., a number of which are mentioned, have been used with a measure of success, but the protection is not quite certain. When the cellulose is but slightly attacked by the molds, treatment with 2% ammonia water or a weak chlorination will remedy the defect. (C)

THE MOTHPROOF FINISHING OF SPUN GOODS AND FABRICS MANUFACTURED FROM MIXTURE OF WOOL AND STAPLE RAYON. Kistermann. *Klepzig's Textil-Z.*, V. 43, P. 219–20, 222, 224 (1940); abs. in *C. A.*, V. 34, Col. 3505 (1940).

Textile fibres of animal origin can be mothproofed by treatment with various grades of Eulan, depending on the fabric and use. The properties

of these products and their application are described in detail and various treatments are discussed fully. (C)

A New Rapid Leasing Device. *Silk J. & Rayon Wld.*, February 1940, V. 16, P. 37.

A Swiss invention designed to increase production and dispense with the present irksome method of section warping. (C)

Physical Properties of Casein and Wool Yarns. W. Davis. *Text. Mfr.*, January 1940, V. 66, P. 16–17.

A comparison of properties of certain wool, casein and wool-casein mixture yarns for knitting. (C)

The Problem of Weaving Spun Rayon Fabrics. Albert Palmer. *Rayon Text. Mo.*, February 1940, V. 21, P. 57–8; March 1940, V. 21, P. 66–8. (C)

Progress in American Deniers and Filament Numbers. H. R. Mauersberger. *Rayon Text. Mo.*, March 1940, V. 21, P. 62–3.

Revised table of deniers and filament numbers of American filament rayon yarns. The last table was published in the September 1939 issue. (C)

Scouring and Milling. D. R. H. Williams. *Text. Mfr.*, January 1940, V. 66, P. 11–12.

Experienced views on the practical side of scouring and milling of worsted goods, in which the possibilities have been neglected, as shown by some new methods invented by the writer. (C)

The Spinning of Spun Rayon Yarns. Carl D. Brandt. *Rayon Text. Mo.*, January 1940, V. 21, P. 71, 72, 79; February 1940, V. 21, P. 53, 54, 68. (C)

Utilization Coefficient of the Strength of the Fibres in Thread. K. I. Koritskii. *Khlopchatobumazhnaya Prom.*, V. 8, No. 1, P. 20–5 (1938); *Chem. Zentr.*, 1938, II, P. 981; abs. in *C. A.*, 1940, V. 34, Col. 4275.

For the calcn. of the percentage strength utilization coeff. of the thread the following formula is given: $B = 0.22n + 0.26 - 4.2p + 23.0$, where n is the no. of fibres in the thread cross section, a the coeff. of twist and p the fibre strength. In mixts. of different kinds of cotton the B coeffs. must be considered in addn. to corresponding fibre lengths. If the B values of 2 different cottons differ by 3% or more (e.g., 45 and 49), then a mixt. of the two is not to be recommended. (C)

"Vinyon"—the New Textile Fibre and Yarn. *Rayon Text. Mo.*, February 1940, V. 21, P. 34–35.

Its origin, characteristic properties, manufacture, uses and applications are described. (C)

III. Chemical and Other Processing (Not Otherwise Classified)

Analysis of Cationic Surface-Active Agents of Trivalent Nitrogen Type. Ralph Hart. *Ind. Eng. Chem.*, July 1940, V. 12, P. 400–402; *Am. Dye. Rptr.*, Aug. 19, 1940, V. 29, p. 411–414.

In spite of the growing application of cationic surface-active agents in the textile and related industries as dye auxiliaries and for finishing, there seems to be a lack of published literature on methods of analysis of such compounds. In this article a procedure is outlined applicable to commercial products of the fatty acid amido-amine type. This procedure includes directions for the determination of the acid used to render the products soluble, nitrogen as alkalinity, total active ingredients or amido-amine, free fatty acids, total fatty acids, condensed amine, and uncondensed amine. (C)

Association between Fibres and Dispersed Particles. C. L. Wall. *Text. Mfr.*, 1940, V. 66, May, P. 202–3, 208; June, P. 245; July, P. 286–7.

I. Energy relationships between fibre aggregates and detergents or dyes, or between dye particles, offer an explanation of a number of known actions in dyeing and treatment of fibrous materials, and are discussed in this paper. II. Discusses the associative properties of soaps, wetting-agents, dyestuffs, etc., arising from the energy relationships of the structure. Some theoretical possibilities in structural types of dyestuffs and detergents are also considered. III. Associative Properties of Ionized Groups. Energy relationships of particles such as dyestuffs in and with water and the effect on associative behavior; affinity of dyestuffs for cellulose or protein in different states of potential energy. (C)

Diffusion of Dyestuff Solutions in Printing Thickeners. Hanns Gerber. *Mell. Textilber.*, V. 20, P. 713–14 (1939); cf. *C. A.*, V. 33, Col. 6054; abs. in *C. A.*, V. 34, Col. 3500 (1940). (C)

Dyeing with Pigments. John T. Gibbons. *Text. Wld.*, July 1940, V. 90, P. 68.

Interest in pigment-dyeing methods has been stimulated by numerous mill experiments carried out during the last year or so on the production on cotton piece goods of solid-color effects with resin-pigment colors and with resin-solvent solutions of ordinary dyes. It is believed, therefore, that many dyers will be interested in the described two old methods of pigment dyeing which have been employed for many years, but which are not widely known in the industry. (C)

Dyeing Rayon Staple Fibre in Raw Stock Form. T. R. Johnson. *Cotton*, July 1940, V. 104, P. 105–109. (C)

Dyeing Yarn and Piece Goods Containing Spun Rayon. Louis I. Fidell. *Cotton*, July 1940, V. 104, P. 100–104. (C)

How to Dye Textiles with Calconese Colors. Louis I. Fidell. *Rayon Text. Mo.*, April 1940, V. 21, P. 79–80; May 1940, V. 21, P. 77–8.

Calconese colors comprise a complete range of dyestuffs, enabling the production of practically all shades on all types of acetate constructions. Their use is described. (C)

THE HYDROPHOBING OF ARTIFICIAL FIBRES AND FABRICS. Richard Flint. *Silk J. & Rayon Wld.*, Oct. 1939, V. 13, P. 808, 834.

The three principal points are (1) The nature of the chemical process of hydrophobing; (2) The manner of change which the properties of artificial fibres are undergoing, and the practical effect of hydrophobing; (3) The method of hydrophobing pieces of cloth. (C)

INCREASING THE WETTING POWER OF MERCERIZING LIQUORS. M. L. Zandberg and M. A. Sagal. *Khlopchato-Bumazhnaya Prom.*, 1939, No. 8–9, P. 63–8; abs. in *C. A.*, V. 34, Col. 3505 (1940). (C)

NEW INVESTIGATIONS ON THE CORRELATION BETWEEN SWELLING AND SOLUBILITY OF DYES IN THICKENING AGENTS. Hanns Gerber. *Mell. Textilber.*, V. 21, P. 76–9 (1940); abs in *C. A.*, V. 34, Col. 3500 (1940). (C)

A NEW THICKENING AGENT FOR TEXTILE PRINTING. F. Nestelberger. *Mell. Textilber.*, V. 21, P. 74–6 (1940); abs. in *C. A.*, V. 34, Col. 3501 (1940).

Colloresin DK, a cellulose base thickener, is coagulable by alkali; this makes possible a new process for vat color printing. The color is printed onto the fabric in a paste of Colloresin DK, aftertreated with alkali and a reducing agent, and finally developed in the usual manner. No loss of color or bleeding occurs at any stage. Colloresin V Extra, non-coagulable by alkali, resembles the natural gums in its general properties. Its advantages and applications with various types of printing colors are discussed and typical recipes are presented. (C)

ONE-BATH DYEING AND DESIZING OF COTTON-RAYON MIXTURES. F. Jahnes. *Mell. Textilber.*, V. 21, P. 73 (1940); abs. in *C. A.*, V. 34, Col. 3501 (1940).

The process consists in passing the material to be treated through a bath contg. Biolase N Extra, an enzymic prepn. The desired direct dyestuff is then added to the bath and the dyeing procedure is carried out. Specific directions are given for cotton and cotton-rayon mixts. (C)

PRETREATMENT OF PILE FABRICS. John C. Kopasz. *Text. Wld.*, July 1940, V. 90, P. 92.

The pretreatment discussed is designed to give uniform and even dyeings, and varies with type of material. (C)

RESEARCH REVIEW ON DIRECT DYEING. Chas. F. Goldthwait. *Rayon Text. Mo.*, V. 21, P. 75–6, 83.

Outlines the current conceptions of the subject, but admits that they fail to provide a complete conception of what takes place. (C)

TEXTILE FINISHING AGENTS. C. J. T. Cronshaw. *Silk J. & Rayon Wld.*, December 1939, V. 16, P. 17–20; January 1940, V. 16, P. 28–9.

Deals with those chemical products—not dyestuffs—all of recent development, which play a part in the finishing or other processing of textile materials. These products, besides being called textile agents, have been called

textile auxiliary products, ancillary products or textile assistants, but none of these is completely satisfactory, largely because they are not classified by any common specific purpose nor fall within a universal chemical category. One thing they do have in common is that they all fulfil some special need in textile manufacture; further they fall within a few chemical types and by their use the textile industry is seeking to add to the inherent qualities of textile materials. (C)

THE TREATMENT OF SPUN RAYONS WITH TEXTONE. G. P. Vincent, A. L. Dubeau and J. W. Ivey. *Am. Dye. Rptr.*, 1940, V. 29, P. P296–P299.

Textone is essentially sodium chlorite, $NaClO_2$. This is a commercially new oxidizing agent and has recently been made available by the Mathieson Alkali Works, Inc. This paper discusses its use for the preparing of spun rayons for bleaching or dyeing. It is claimed to reduce processing time and to obtain a superior result at no additional cost and very often at a reduced cost. (C)

VINYL RESINS AND HOW THEY ARE USED. J. Wakelin. *Text. Col.*, August 1939, V. 61, P. 517. (C)

WEAVING AND DYEING OF SILK AND ACETATE RAYON: CERTAIN DIFFICULTIES IN. W. Weltzien. *Zellwolle, Kunstseide, Seide*, 1939, V. 44, P. 378–81; abs. in *C. A.*, 1940, V. 34, Col. 1489.

A detailed discussion of processing difficulties in strand-dyed Organzin as well as in the degumming of silk in the presence of acetate rayon. Chem. and mech. methods of overcoming these difficulties are described. (C)

WETTING AND WETTING AGENTS; WASHING, SCOURING, CLEANSING AND OTHER DETERGENT AGENTS AND PROCESSES; EMULSIFYING, EMULSIFIERS AND EMULSIONS; THEIR MANUFACTURE, TESTING AND USE: A Bibliography of the Literature for 1937. Chas. E. Mullin. *Text. Col.*, August 1939, P. 519–21; September 1939, P. 595–8; October, P. 662–6. (C)

WOOL: ACTION OF ALKALIES ON. E. Schmidt. *Teintex*, 1939, V. 4, P. 563–6; abs. in *C. A.*, 1940, V. 34, Col. 2181.

Treating wool with alk. solns. produces three effects: (a) the wool absorbs a certain amt. of the alkali, (b) more or less swelling occurs, (c) the keratin mol. is hydrolyzed. Hydrolysis occurs more readily in alk. than in acid soln. Swelling of wool increases with the pH. The action of the following substances is discussed: caustic alkali, alk. earths, NH_3, Schweitzer's soln., org. bases, alk. carbonates, borax, Na_3PO_4, silicates and soaps. (C)

WOOL: PROTECTION OF—DURING REDYEING. W. Pflumm. *Klepzig's Textil-Z.*, 1939, V. 42, P. 684–5; abs. in *C. A.*, 1940, V. 34, Col. 1489.

Good protective agents are proteins related to those in wool (*e.g.*, bone glue), protein degradation products, sulfite liquors of the type used in mordant treatments and Cr dyeings, and "Modinal." The latter product has good leveling and reviving properties and improves the phys. qualities of wool. Three photomicrographs are shown. (C)

WOOLEN PIECE GOODS: THE PROCESSING OF EMBOSSED EFFECTS ON. S. Wouble. *Text. Col.*, August 1939, V. 61, P. 551. (C)

YARNS: DISCONTINUOUS COLORING. P. Merey. *Teintex*, 1940, V. 4, P. 588–591; abs. in *J. T. I.*, May 1940, P. A252.

Dyeing methods and apparatus for the production of multi-color, mottled, shaded and other irregular color effects on cotton, rayon, wool, silk and mixture yarns are described. (C)

IV. RESEARCH METHODS AND APPARATUS

AN ACHROMATIC OBJECTIVE FOR USE IN ULTRAVIOLET MICROSCOPY. B. K. Johnson. *Proc. Phys. Soc.*, 1939, V. 51, No. 288, P. 1034–39; abs. in *Eastman Kodak Abs. Bull.*, May 1940, P. 278.

A microscope objective is described having a 25-mm. focal length and a numerical aperture of 0.20. It is a cemented doublet made of fused quartz and lithium fluoride and is achromatized for wave lengths of 5461 A. and 2749 A. The secondary spectrum is very small. Tests made on the objective are described. Ultraviolet filters with a high-pressure mercury lamp are used in the illuminating system. (S)

ANALYSIS OF CELLULOSE AND BLEACHING METHODS FOR CELLULOSE MATERIALS. II. Application of a New Method of Cellulose Estimation to Various Plant Analyses. Sin-iti Honda. *J. Agr. Chem. Soc. Japan*, V. 15, P. 1111–17; *Bull. Agr. Chem. Soc. Japan*, V. 15, P. 150–1 (in English) (1939); cf. *C. A.*, V. 34, Col. 1479; abs. in *C. A.*, V. 34, Col. 3489 (1940).

In the bleaching powder method a higher value for total cellulose and α-cellulose was obtained. This method was found to be simpler and more rapid than the Cross and Bevan method (cf. *C. A.*, V. 1, Col. 2489). (C)

COMPRESSION TESTS ON RAYON FLOCK. Erwin J. Saxl. *Text. Rsch.*, August 1940, V. 10, P. 425–432; *Rayon Text. Mo.*, June 1940, V. 21, P. 56–8; July 1940, V. 21, P. 71–2. (C)

DELUSTRED RAYON: DETERMINATION OF NATURE OF DELUSTRING TREATMENT. *Kunstseide u. Zellwolle*, 1940, V. 22, P. 80–4; abs. in *J. T. I.*, June 1940, V. 31, P. A335. (C)

THE ESTIMATION OF CELLULOSE DEGRADATION. H. Rath and H. Rademacher. *Mell. Textilber.*, V. 20, P. 749–51 (1939); abs. in *C. A.*, V. 34, Col. 3491 (1940).

The value of Cu no., Ag no., methylene blue no., etc., as indexes of the state of degradation of a cellulose fibre depends on the answer to 3 questions: (1) Can the chemically active portion be sepd. by a previous alkali treatment? (2) Can the reactive end groups be of a differing nature? (3) Can groups other than the end groups affect the detn. reaction? Samples of staple rayon were extd. with boiling alk. solns. varying in concn. from 0.2% Na_2CO_3 to 5.0% NaOH and the Cu no. was detd. after the treatment. The Cu no. progressively decreased and the reducing power of the alk. ext. correspondingly increased with increasing concn. of alkali. The methylene blue no. and the Ag no. showed that carboxyl end groups as well as aldehyde end groups are active in these detns. and their effect must be taken into account. Studies of the degree of heterogeneity of regenerated cellulose samples indicated that degradation may take place by oxidative

fission in the middle of the chain as well as by hydrolysis at the end groups. (C)

INFRARED PHOTOGRAPHY OF BLACK-DYED MATERIALS. P. W. Danckwortt. *Z. angew. Phot.*, April 1939, V. 1, No. 2, P. 48–50; abs. in *Eastman Kodak Abs. Bull.*, May 1940, P. 247.

A study of interest to textile manufacturers as well as to criminologists was made. Using Agfa Infrared film and an Agfa No. 85 infrared filter, the photographic reflectivities of a large number of black dyes (I. G. Farbenind. A.-G. acid and chrome dyes) which had been impregnated in cloth were measured. Thirty-six exposures were made on 12 films. Details of making these measurements are given. A table of over 200 dyes is included. (S)

THE KAYSER TORSOMETER. *Rayon Text. Mo.*, April 1940, V. 21, P. 97.

The Torsometer was developed to measure torque and to determine the control torsion in yarns after processing, such as soaking, twisting and setting, also to serve as a guide in developing new combination yarns and aid in regulating the temperature and relative humidity in vapor cabinets. The Torsometer measures the torsion of a twisted yarn, which is a force that tends to return the yarn to a state of rest. This force causes the kinking of yarns and the twisting of silk hosiery. (C)

PHYSICAL AND CHEMICAL METHODS FOR TESTING STAPLE RAYON. Walther Schieber. *Mell. Textilber.*, 1939, V. 20, P. 617–25; *Angew. Chem.*, 1939, V. 52, P. 561–8; cf. *C. A.*, V. 34, 1181; abs. in *C. A.*, 1940, V. 34, Col. 3096.

Undue emphasis has been laid on wet and dry tensile strength as an index of the quality of staple rayon. Material of high tensile strength may have other characteristics which render it unfit for general use. Phys. tests which should be applied to completely evaluate the fibre are: fastness to knotting, wet and dry tensile strength, wet and dry total elongation, elastic elongation, relative wet strength, fastness to creasing, retention of flexibility, work required for elongation to the elastic limit, work required for elongation to the breaking point. Chem. tests which should be applied are: titer, av. polymerization no., alkali soly., area under the Dolmetsch soly. diagram, orientation and packing d. Quant. relations among these properties are presented for a no. of com. fibres. The bearing of each phys. and chem. property considered on working characteristics, wearing quality, and resistance to washing and bleaching is thoroughly discussed. These tests are especially important and valuable in developing new types of rayon and fibres for special purposes. (C)

THE QUANTITATIVE DETERMINATION OF CASEIN FIBRE CONTENT OF MIXED GOODS. K. Hartung. *Mell. Textilber.*, 1939, V. 20, P. 697–8; abs. in *C. A.*, 1940, V. 34, Col. 3097.

Casein fibres are attached and rendered sol. by pepsin and HCl, provided their HCHO content has been previously removed by treatment with H_2SO_4. Wool and other fibres are unaltered by this treatment. The method of analysis, applicable to dyed or undyed material, is as follows: a 5 g. sample is extd. with ether in a Soxhlet app., dried at 105°, and weighed.

It is then boiled ½ hr. in 200 cc. of 0.1–0.2% H_2SO_4, the sol. being maintained by addns. of hot water. After thorough rinsing it is added to a soln. of 2 g. pepsin and 0.5 cc. concd. HCl in 100 cc. water, and kept at 60° for 3 hrs. with occasional stirring. It is then rinsed in cold water, dried and weighed. A brief review of previous qual. and quant. methods is given. (C)

THE QUANTITATIVE SEPARATION OF SHEEP WOOL AND CASEIN WOOL. P. A. Koch. *Klepzig's Textil-Z.*, V. 43, P. 248–9 (1940); abs in *C. A.*, V. 34, Col. 3503.

A discussion of various procedures. 7 references. (C)

TESTING JAPAPESE RAW SILK. *Silk J. & Rayon Wld.*, December 1939, V. 16, P. 15–6.

All Japanese raw silk intended for export must first be tested at the Government Conditioning House in either Yokohama or Kobe. The test is divided into two parts: (a) a quality test comprising visual inspection and mechanical testing, and (b) a conditioned weight test. Certificates are issued which show the testing results at a glance and these form the basis of subsèquent business transactions. (C)

TESTING OF STAPLE RAYON, WITH ESPECIAL ATTENTION TO ALTERATIONS IN PHYSICAL PROPERTIES WHICH ARE CAUSED BY STRETCHING. R. Stoll and E. Rall. *Mell. Textilber.*, V. 20, P. 734–7 (1939); abs. in *C. A.*, V. 34, Col. 3503 (1940).

The wearing quality of finished material made from natural fibres is usually directly proportional to the tensile strength of the individual fibres. With mercerized cotton and staple rayon, however, increased tensile strength is accompanied by decreased elasticity and stretching power. This latter factor means a lowered resistance of the fibre to the crosswise forces brought into play by spinning and weaving, engendering poorer wearing quality. Cross forces on the fibre are therefore fully as important as longitudinal forces in the evaluation of staple rayon. The resistance to knotting of a fibre is a good measure of its resistance to cross forces, and should be used in conjunction with tensile strength tests. There is no direct relationship between these properties because of anisotropy of the fibre. (C)

TEXTILE FIBRES: MICROSCOPIC EXAMINATION. P. Tissot. *TIBA*, 1940, V. 18, P. 43–9; abs. *J. T. I.*, April 1940, P. A203.

Microscopic methods for the examination of textile fibres are discussed and tests for the identification of animal casein and wild silk fibres, the detection of damaged wool, the identification of cotton, Manilla hemp, jute, ramie and the various types of rayon, and for the differentiation of hemp from linen and Manilla hemp from coir fibre are described. The preparation of the reagents is briefly described. (C)

WOOL-CASEIN FIBRE MIXTURES: QUANTITATIVE ANALYSIS. P. A. Koch. *Klepzig's Textil.-Z.*, 1940, V. 43, P. 248–9; abs. in *J. T. I.*, May 1940, P. A265.

Various methods for the quantitative analysis of mixtures of wool and casein fibres, including a microscopic method involving counting fibres in

gelatin, methods depending on solution of the wool in caustic soda solution, a method depending on solution of the casein fibres in a hydrchloric acid pepsin solution, and a method involving carbonization with aluminium chloride solution, are briefly described. (C)

V. Pure Science, Economics and Misc.

Collagen Fibres: Molecular Structure. W. T. Astbury and Florence O. Bell. *Nature*, 1940, V. 145, P. 421–2; abs. in *J. T. I.*, May 1940, P. A284.

A model for the molecular structure of fibres of the collagen group is shown and recent chemical and X-ray evidence on which it is based is briefly discussed. The average length per amino-acid residue in the direction of the fibre axis is about 2.85 A., and the intramolecular pattern requires no less than 288 residues in a row. Owing to the high content of proline and hydroxyproline the polypeptide chains are partly in a *cis*-configuration. Among other points in its favor, the model provides an explanation of the fact that, although the collagen configuration is shorter than that of the β-proteins, it is practically inextensible. All the extended forms of the fibrous proteins are built from polypeptide chains in either the *cis*- or the *trans*-configuration. (C)

How to Make Money Make More Money through Research. Fuller E. Callaway, Jr. *Text. Wld.*, February 1940, V. 90, P. 98–9.

The author concludes his article as follows: "I have already said that if all the textile mills of the country would pay into a common fund a definite percentage of their net invoices, we would have a sum that would compare favorably with any other industry. With this we could learn about the fibres with which we work, produce new materials, develop new processes, contrive new machines, and make new and hitherto unimagined yarn and fabric applications. We could coordinate the activities of research agencies now existing, stimulate the establishment of research consciousness and activity in units where it is now unknown, develop cooperation among the various agencies and units, and carry on a continuous program of expansion. If this is done, I believe we shall continue to have a textile industry. If this is not done, there will doubtless continue to be a textile industry, but it may not continue to be ours." (C)

Hygienic and Technical Aspects of Water Repellent Clothing. Leo Stein. *Am. Dye. Rptr.*, 1940, V. 29, P. 352–355. (C)

The Intelligent Use of Numerical Aperture. L. V. Foster. *J. Biol. Phot. Assoc.*, December 1939, V. 8, P. 60–8; abs. in *Eastman Kodak Abs. Bull.*, April 1940, P. 178.

A simplified explanation of the resolving power of a microscope is given, using diagrams of diffraction gratings for illustrations. The use of oblique illumination to include higher orders of the diffraction spectrum than can be done by central illumination is explained. Diagrams of the Abbe condenser, an aplanatic condenser, and an achromatic condenser are given and their merits discussed. (S)